1986

ANNUAL QUALITY ASSURANCE PERFORMANCE REPORT

SECTION 8

SEDIMENT AND SOIL SAMPLES

INORGANIC TRACE CONTAMINANTS SECTION

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Laboratory Services Branch
Ministry of the Environment

1986

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SECTION 8

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Inorganic Trace Contaminants Section
Laboratory Services Branch
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INORGANIC TRACE CONTAMINANTS SECTION

SUMMARY

I. Introduction

The Inorganic Trace Contaminants Section of the Ministry of the Environment, Laboratory Services Branch is responsible for the analysis of a wide variety of sample types for metals and non-metals. The use of sensitive instrumentation and methodologies appropriate to the sample matrix, combined with quality assurance programs, ensures that the Section is able to maintain a high standard of analytical performance. This performance is monitored through regular internal quality control and assurance programs as well as participation in interlaboratory round-robins. This QA report summarizes the methodologies used for analysis of these samples and the supporting internal quality assurance data.

This report is assembled in sections that reflect the analyses performed on different sample matrices in support of the programs of the Ministry of the Environment. Coincidentally, these divisions also reflect the supervisory responsibilities within the Section.

II. Quality Control and Assurance

The objectives of the quality control and assurance programs are to ensure that all of the components of the analytical process are under control and to ensure immediate detection and correction of unacceptable analytical performance. The program monitors all of the reagents, instrumentation, calibration and recovery components of the analytical system.

A. Quality Control

Quality control of the analytical process takes place at the instrument level and is intended to ensure that the instrumentation is operating according to established criteria. This control function ensures that instrument calibration, standardization, slope and intercept, and instrumental drift meet these criteria.

B. Quality Assurance

Quality assurance of the analytical process takes place after the results have been generated and is intended to ensure that the analytical protocols of sample preparation and digestion have been carried out correctly. This control function ensures that reagent blanks, digested standards, sample duplicates and recovery materials meet established response criteria.

III. Report Format

The report consists of one page method summaries and one page data summaries of blanks, between-run controls and within-run duplicates in formats that are common to all of the parameter/matrix combinations. The method summaries give a brief outline of the sample preparation and measurement procedures. The data summaries consist of annual mean values with standard deviations.

For the within-run duplicates, the data set is subdivided into ranges approximating 0 to 20 %, 20 to 50 % and 50 to 100% of the analytical range. All results for duplicates reported to the data base that are "<" or that have been diluted into the range are excluded from the statistical analysis.

The standard deviations for blanks and between-run controls are calculated using formula I. Formula II is used for the calculations for within-run duplicates.

$$sd = sqrt[\{(sumx2 - (sumx)2\}/n/(n-1)] \quad .....I$$

$$sd = sqrt(sumd2/2n) \quad .....II$$

where : x = the individual values; n = the number of events
d = the differences between pairs of duplicates

The data is stored in a personal computer using BMB Manager II files. All data manipulations, reports generated etc, are performed using applications written in Manager Math.

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8. Sediment and Soil

8.1 Sediment Samples

Sediment samples are samples collected from the bottoms of bodies of water and are normally in a water saturated state when taken. All sediment samples are dried and ground to a uniform particle size prior to analysis. Materials that cannot be pulverized or that may not reflect the intent of the sampling program (stones or whole shells for example) are removed by hand prior to sample preparation. In general, only those materials that pass a 20 mesh sieve (1 mm) are analysed.

8.2 Soil Samples

Soil samples are collected from the surdace of the earth and are normally unsaturated with respect to water content. These samples are prepared much the same as sediment samples.

Table 8.1 summarizes the parameters determined, the preparation methods and the instrument types used for the analysis sediment and soil samples.

TABLE 8.1

Parameter	Collection Device	Preparation	Analysis
Metals	Plastic or glass jars	Acid Digest	AAS
Anions	Plastic or glass jars	Ext/Filt	IC
Cyanide	Plastic or glass jars	Distillation	Colorimetry
Hydrides	Plastic or glass jars	Acid Digest	AAS
Total N&P	Plastic or Glass jars	Acid digest	Colorimetry
Mercury	Plastic or glass jars	Acid digest	Cold Vapour AAS

8.3 Sediment and Soil Quality Assurance

Sub aliquots of sediment or soil samples are analysed separately to generate duplicate results. Blanks will consist of the digestion acid or distilled water as appropriate.

Table 8.2 summarizes the QA materials used when sediment samples are being analysed.

TABLE 8.2

Sample Designation	Type	Parameter
qca,qcb	Standard solution	Cyanide
qcr	Standard solution (0.2 mg/L)	Cyanide
QCRS85,RS85-1,refa soilqc,S85-1	composite soil (prep 1985)	AAS Metals
soil cont1,2	Composite soil	Hydride Metals
veg contro	Composite vegetation (used with veg as well)	Hydride metals
soil	Composite soil	Chloride
clay1,2	composite clay soils	Chloride
sedqc	composite sediment	Sulfate
orch leaves	NBS orchard leaves 1571	Hydride metals,N,P
con 684	Composite soil sample	Mercury
BACO3	Barium Carbonate	Carbonate
CACO3	Calcium Carbonate	Carbonate

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: ALUMINUM TEST CODE: ALUT SAMPLE TYPE: SEDIMENT
UNIT: Vegetation/Soil/Sediment SUPERVISOR: L. Pastorek

METHOD CODE:533AA0 HMARSOIL - ICP/AES
REVISION NO: Original DATE: 1980
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- 10 to 20 g of wet sample
Container- 500 ml PET jar
Preservative- None
Other- Samples are air-dried and ground to less than 45 mesh.

SAMPLE PREPARATION:Partial Extn.-Yes Total Extn.- % Extracted-34±10
Procedure- Weigh 0.500g sample into a calibrated digestion tube and add 1.5 ml HNO_3 . Mix and stand at room temperature for 2 hrs. Add 4.5 ml HCl to each tube, mix and stand at room temperature for another hour. Digest in a hot block for 1 hour at 40°C, 1 hour at 60°C, 1 hour at 80°C and 4 hrs at 100°C allowing 1/2 hr. ramping times
NOTE: The final volume should be less than 2 ml; if more, continue heating at 125°C to accomplish this.
Dilute with DDW to 25 ml and mix thoroughly.
Settle out suspended particles overnight and decant clear supernatant into disposable plastic tubes for analysis by AES-ICP. Samples requiring 5 or more metals are analyzed by AES-ICP (see below).
INTERFERENCES: Several, which are compensated for by the computer program.
REPORTING RESULTS: Two significant figures. µg/g Al
INSTRUMENTATION: For the determination of 5 or more elements:
Inductively coupled Plasma Emission Spectrometer- Jobin-Yvon 1 m.JY48P with Autosampler & DEC computer.PET interface to LIS.(See "Remarks").
Calibration Range: 0 - 50 mg/l.(JA)
Resolution: Four significant figures (0.0001 mg/l).
Sensitivity: Not applicable (NA).
Instrument Detection Limit:.08 mg/l (JY).
PERFORMANCE CHARACTERISTICS:
Routine Operating Range-JY=4.0 to 25000 µg/g
Accuracy- Not determined (ND).
Precision of Controls-

	A	B
mean	11000µg/g	
std. dev.	560 µg/g	
R.S.D.	5.2 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	120	440	1000
mean	3400	9500	17000

W .2 µg/g T 1.0 µg/g

CONTROL LIMITS: µg/g

Control		W.L. (x ±2σ)	R.L. (x ±3σ)
QCRS85-1	L.L.	9900	9300
	U.L.	12000	13000

REMARKS:- Determination of boron MUST be on Atom Scan 2400 only.
- Extraction % (±) based on average recovery of 4 samples from Soil Research Institute - SO1, SO2, SO3 & SO4.
- Samples requiring only Mg, Al and/or Ca are analyzed by Atom Scan.

SUMMARY REPORT OF QUALITY CONTROL DATA

ALUMINUM IN SEDIMENT

Operating Range = 4.000 to 25000. ug/g

IN - RUN DUPLICATES

Range	<4.000	4.000 to5000.0	5000.0to12500.	12500.to25000.	>25000.
no.	1	9	16	17	1
s.w.		116.4420	444.9660	1000.947	
mean		3366.230	9481.290	16618.51	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
QCRS85-1	118	10692.91	557.5620	5.21

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
rb	62	4.845	3.1735

DATE 87/04/22

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME:Antimony TEST CODE:SBUT SAMPLE TYPE:SEDIMENT
UNIT: Biomaterials SUPERVISOR: R. S. Sadana

METHOD CODE:510EF3 TYPE: Semi-aut. hydr. gen - flameless AAS
REVISION NO: Original DATE: January, 1983
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- 10 to 20 g of wet sample
Container- 500ml PET jar
Preservative- None
Other- Samples are air-dried and ground to less than 45 mesh

SAMPLE PREPARATION:Partial Extn.- Total Extn.-Yes % Extracted->90
Procedure- Weigh 60 mg (45 mesh) sample into a 18 x 150 mm pyrex graduated test tube. Add 3 ml of acid mixture (6 nitric: 3 sulphuric: 1 perchloric). Process in batches of 80 samples including blanks, calibration standards and controls.
Digest in an aluminum hot block at a medium setting on the hot plate for 14 hrs until dense white fumes appear.
Cool,add 0.5 ml of distilled water, then add 2 ml conc. HCl.
Dilute to 15 ml with distilled water and mix.
Feed the prepared solutions to the automated system for the determination of antimony by the hydride-FAAS technique.
INTERFERENCES: Excessive concentrations of Cu, Fe and Ni may interfere.
REPORTING RESULTS:2 dec. <10, 1 dec. <100, 0 dec. if >100 µg/g
INSTRUMENTATION: Atomic absorption spectrophotometer (Varian Techtron 1200 & AA-5,with chart recorder, peristaltic pump and autosampler.
Open ended heated quartz "T" cell; gas-liquid separator.
Calibration Range:0 - 40 ng/ml (linear<20ng/ml)
Resolution: 0.01 absorbance (unexpanded scale)
Sensitivity:0.02 µg/ml reads 0.15 abs.
Instrument Detection Limit: 0.001 µg/ml
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.3 to 10 ug/g
Accuracy- 99% (NBS orchard leaves)
Precision of Controls-

	A	B
mean	.55 µg/g	18 µg/g
std. dev.	.49 µg/g	3.5 µg/g
R.S.D.	89 %	19 %

Precision of Duplicates-low range mid range high range
s.d. .014
mean .41
W 0.2 µg/g T 1.0 µg/g

CONTROL LIMITS:

REMARKS:

- Detection Limit - 3x std. dev. of low range within-run duplicates.
- Accuracy - Ratio of mean to cert. value in ref. mat. (%).

SUMMARY REPORT OF QUALITY CONTROL DATA

ANTIMONY IN SEDIMENT

Operating Range = 0.300 to 10.0 ug/g

IN - RUN DUPLICATES

Range	<0.300	0.300 to2.00	2.00 to5.00	5.00 to10.0	>10.0
no.	0	1	0	0	0
s.w.		0.0141	0.0000	0.0000	
mean		0.4100	0.0000	0.0000	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
orch leave	6	2.855	0.4465	15.64
veg contro	14	0.484	0.1798	37.15
soil cont1	34	0.554	0.4929	88.97
soil cont2	26	18.446	3.4589	18.75

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLK	0	0.000	0.0000

DATE 87/03/17

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME:Arsenic TEST CODE:ASUT SAMPLE TYPE:SEDIMENT
UNIT: Biomaterials SUPERVISOR: R. S. Sadana

METHOD CODE:510EF3 TYPE: Semi-aut. hydr. gen - flameless AAS
REVISION NO: Original DATE: January, 1983
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- 10 to 20 g of wet sample
Container- 500 ml PET jar
Preservative- None
Other- Samples are air-dried and ground to less than 45 mesh

SAMPLE PREPARATION:Partial Extn.- Total Extn.-Yes % Extracted->90
Procedure- Weigh 60 mg (45 mesh) sample into a 18 x 150 mm pyrex graduated test tube. Add 3 ml of acid mixture (6 nitric: 3 sulphuric: 1 perchloric). Process in batches of 80 samples including blanks, calibration standards and controls.
Digest in an aluminum hot block at a medium setting on the hot plate for 14 hrs until dense white fumes appear.
Cool,add 0.5 ml of distilled water, then add 2 ml conc. HCl.
Dilute to 15 ml with distilled water and mix.
Feed the prepared solutions to the automated system for the determination of arsenic by the hydride-FAAS technique.
INTERFERENCES: Excessive concentrations of Cu, Fe and Ni may interfere.
REPORTING RESULTS:2 dec. <10, 1 dec. <100, 0 dec. if >100 µg/g.
INSTRUMENTATION: Atomic absorption spectrophotometer (Varian Techtron 1200 & AA-5,with chart recorder, peristaltic pump and autosampler.
Open ended heated quartz "T" cell; gas-liquid separator
Calibration Range:0 - 40 ng/ml (linear<20ng/ml)
Resolution: 0.01 absorbance (unexpanded scale)
Sensitivity:0.02 µg/ml reads 0.15 abs.
Instrument Detection Limit: 0.001 µg/ml
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.3 to 10 µg/g
Accuracy- 99% (NBS orchard leaves)
Precision of Controls-

	A	B
mean	5.6 µg/g	12.2 µg/g
std. dev.	.81	1.58
R.S.D.	14 %	13%

Precision of Duplicates-

	low range	mid range	high range
s.d.	.14	.37	.38
mean	1.2	3.6	7.8

W 0.2 µg/g T 1.0 µg/g

CONTROL LIMITS:

REMARKS:

- Detection Limit - 3x std. dev. of low range within-run duplicates.
- Accuracy - Ratio of mean to cert. value in ref. mat. (%).

SUMMARY REPORT OF QUALITY CONTROL DATA

ARSENIC IN SEDIMENT

Operating Range = 0.300 to 10.0 ug/g

IN - RUN DUPLICATES

Range	<0.300	0.300 to2.00	2.00 to5.00	5.00 to10.0	>10.0
no.	0	3	6	6	3
s.w.		0.1420	0.3654	0.3819	
mean		1.1750	3.5590	7.7920	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
orch leave	45	9.297	1.0512	11.31
veg contro	68	9.184	1.4021	15.27
soil cont1	120	5.627	0.8063	14.33
soil cont2	104	12.216	1.5785	12.92

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLK	0	0.000	0.0000

DATE 87/03/17

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: BARIUM TEST CODE: BAUT SAMPLE TYPE: SEDIMENT
UNIT: Vegetation/Soil/Sediment SUPERVISOR: L. Pastorek

METHOD CODE:533AA0 HMARSOIL - ICP/AES
REVISION NO: Original DATE: 1980
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- 10 to 20 g of wet sample
Container- 500 ml PET jar
Preservative- None
Other- Samples are air-dried and ground to less than 45 mesh.

SAMPLE PREPARATION:Partial Extn.-Yes Total Extn.- % Extracted-ND
Procedure- Weigh 0.500g sample into a calibrated digestion tube and add 1.5 ml HNO3 . Mix and stand at room temperature for 2 hrs. Add 4.5 ml HCl to each tube, mix and stand at room temperature for another hour. Digest in a hot block for 1 hour at 40°C, 1 hour at 60°C, 1 hour at 80°C and 4 hrs at 100°C allowing 1/2 hr. ramping times
NOTE: The final volume should be less than 2 ml; if more, continue heating at 125°C to accomplish this.
Dilute with DDW to 25 ml and mix thoroughly.
Settle out suspended particles overnight and decant clear supernatant into disposable plastic tubes for analysis by AES-ICP. Samples requiring 5 or more metals are analyzed by AES-ICP (see below).
INTERFERENCES: Several, which are compensated for by the computer program.
REPORTING RESULTS: Two significant figures. µg/g Ba
INSTRUMENTATION: For the determination of 5 or more elements:
Inductively coupled Plasma Emission Spectrometer- Jobin-Yvon 1 m.JY48P with Autosampler & DEC computer.PET interface to LIS.(See "Remarks").
Calibration Range: 0 - 10 mg/l.
Resolution: Four significant figures (0.0001 mg/l).
Sensitivity: Not applicable (NA).
Instrument Detection Limit:.002 mg/l.
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.1 to 100 µg/g.
Accuracy- Not determined (ND).
Precision of Controls-

	A	B
mean	81 µg/g	
std. dev.	4.7 µg/g	
R.S.D.	5.8 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	0.46	2.8	1.7
mean	14	33	72

W 0.5 µg/g T 2.5 µg/g

CONTROL LIMITS: µg/g

Control		W.L. (x ±2σ)	R.L. (x ±3σ)
QCRS85-1	L.L.	72	67
	U.L.	90	95

REMARKS:- Determination of boron MUST be on Atom Scan 2400 only.
- Extraction % (±) based on average recovery of 4 samples from Soil Research Institute - SO1, SO2, SO3 & SO4.
- Samples requiring only Mg, Al and/or Ca are analyzed by Atom Scan.

SUMMARY REPORT OF QUALITY CONTROL DATA

BARIUM IN SEDIMENT

Operating Range = 0.100 to 100.0 ug/g

IN - RUN DUPLICATES

Range	<0.100	0.100 to20.00	20.00 to50.00	50.00 to100.0	>100.0
no.	6	8	9	10	11
s.w.		0.4601	2.8454	1.7414	
mean		13.8078	33.0999	72.3438	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
QCRS85-1	121	81.517	4.6937	5.76

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
rb	48	0.160	0.3145

DATE 87/04/23

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: BERYLLIUM TEST CODE: BEUT SAMPLE TYPE: SEDIMENT
UNIT: Vegetation/Soil/Sediment SUPERVISOR: L. Pastorek

METHOD CODE:535AA0 HMARSOIL - ICP/AES
REVISION NO: Original DATE: 1980
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- 10 to 20 g wet sample
Container- 500 ml PET jar
Preservative- None
Other- Samples are air-dried and ground to less than 45 mesh.

SAMPLE PREPARATION:Partial Extn.-Yes Total Extn.- % Extracted-ND
Procedure- Weigh 0.500g sample into a calibrated digestion tube and add 1.5 ml HNO3 . Mix and stand at room temperature for 2 hrs. Add 4.5 ml HCl to each tube, mix and stand at room temperature for another hour. Digest in a hot block for 1 hour at 40°C, 1 hour at 60°C, 1 hour at 80°C and 4 hrs at 100°C allowing 1/2 hr. ramping times
NOTE: The final volume should be less than 2 ml; if more, continue heating at 125°C to accomplish this.
Dilute with DDW to 25 ml and mix thoroughly.
Settle out suspended particles overnight and decant clear supernatant into disposable plastic tubes for analysis by AES-ICP. Samples requiring 5 or more metals are analyzed by AES-ICP (see below).
INTERFERENCES: Several, which are compensated for by the computer program.
REPORTING RESULTS: Two significant figures. µg/g Be
INSTRUMENTATION: For the determination of 5 or more elements:
Inductively coupled Plasma Emission Spectrometer- Jobin-Yvon 1 m.JY48P with Autosampler & DEC computer.PET interface to LIS.(See "Remarks").
Calibration Range: 0 - 10 mg/l.
Resolution: Four significant figures (0.0001 mg/l).
Sensitivity: Not applicable (NA).
Instrument Detection Limit: .003 mg/l.
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.2 to 10 µg/g.
Accuracy- Not determined (ND).
Precision of Controls-

	A	B
mean	1.1 µg/g	
std. dev.	.096 µg/g	
R.S.D.	8.5 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	ND	ND	ND
mean	ND	ND	ND

W 0.2 µg/g T 1.0 µg/g

CONTROL LIMITS: µg/g

Control		W.L. (x ±2σ)	R.L. (x ±3σ)
QCRS85-1	L.L.	0.91	0.81
	U.L.	1.3	1.4

REMARKS:- Determination of boron MUST be on Atom Scan 2400 only.
- Extraction % (±) based on average recovery of 4 samples from Soil Research Institute - SO1, SO2, SO3 & SO4.
- Samples requiring only Mg, Al and/or Ca are analyzed by Atom Scan.

SUMMARY REPORT OF QUALITY CONTROL DATA

BERYLLIUM IN SEDIMENT

Operating Range = 0.200 to 10.0 ug/g

IN - RUN DUPLICATES

Range	<0.200	0.200 to2.00	2.00 to5.00	5.00 to10.0	>10.0
no.	27	17	0	0	0
s.w.		0.3279	0.0000	0.0000	
mean		1.1428	0.0000	0.0000	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
QCRS85-1	9	1.130	0.0958	8.48

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
rb	37	0.045	0.0315

DATE 87/04/23

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: BORON (OLD) TEST CODE: BBUT SAMPLE TYPE: SEDIMENT
UNIT: Vegetation/Soil/Sediment SUPERVISOR: L. Pastorek

METHOD CODE: HMARSOIL - ICP/AES
REVISION NO: Original DATE: 1980
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- 10 to 20 g wet sample
Container- 500 ml PET jar
Preservative- None
Other- Samples are air-dried and ground to less than 45 mesh.

SAMPLE PREPARATION:Partial Extn.-Yes Total Extn.- % Extracted-ND
Procedure- Weigh 0.500g sample into a calibrated digestion tube and add 1.5 ml HNO_3 . Mix and stand at room temperature for 2 hrs. Add 4.5 ml HCl to each tube, mix and stand at room temperature for another hour. Digest in a hot block for 1 hour at 40C, 1 hour at 60C, 1 hour at 80C and 4 hrs at 100C (allowing 1/2 hr. ramping times)
NOTE: The final volume should be less than 2 ml; if more, continue heating at 125C to accomplish this.
Dilute with DDW to 25 ml and mix thoroughly.
Settle out suspended particles overnight and decant clear supernatant into disposable plastic tubes for analysis by AES-ICP. Samples requiring 5 or more metals are analyzed by AES-ICP (see below).
INTERFERENCES: Several, which are compensated for by the computer program.
REPORTING RESULTS: Two significant figures.
INSTRUMENTATION: For the determination of 5 or more elements:
Inductively coupled Plasma Emission Spectrometer- Jobin-Yvon 1 m.JY48P with Autosampler & DEC computer.PET interface to LIS.(See "Remarks").
Calibration Range: 0 -10 mg/l.(JA)
Resolution: Four significant figures (0.0001 mg/l).
Sensitivity: Not applicable (NA).
Instrument Detection Limit:.07 mg/l.
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 3.5 to 100 µg/g.
Accuracy- Not determined (ND).
Precision of Controls-

	A	B
mean	12 µg/g	
std. dev.	5.4µg/g	
R.S.D.	45 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	2.8	1.5	
mean	10	32	

W 2 µg/g T 10 µg/g

CONTROL LIMITS: µg/g		W.L. (x ±2σ)	R.L. (x ±3σ)
Control	L.L.	1.2	0
QCRS85-1	U.L.	23	28

REMARKS:- Determination of boron MUST be on Atom Scan 2400 only.
- Extraction % (±)- ND. - based on average recovery of 4 samples from Soil Research Institute - SO1, SO2, SO3 & SO4.

SUMMARY REPORT OF QUALITY CONTROL DATA

BORON-ɔy IN SEDIMENT

Operating Range = 3.500 to 100.0 ug/g

IN - RUN DUPLICATES

Range	<3.500	3.500 to20.00	20.00 to50.00	50.00 to100.0	>100.0
no.	22	15	5	2	0
s.w.		2.9728	1.5496	2.8651	
mean		11.1239	32.0552	55.5380	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
QCRS85-1	105	12.037	5.4059	44.91

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
rb	100	3.680	2.5430

DATE 87/04/23

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: CALCIUM TEST CODE: CAUT SAMPLE TYPE: SEDIMENT
UNIT: Vegetation/Soil/Sediment SUPERVISOR: L. Pastorek

METHOD CODE:535AA0 HMARSOIL - ICP/AES
REVISION NO: Original DATE: 1980
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- 10 to 20 g of wet sample
Container- 500 ml PET jar.
Preservative- None
Other- Samples are air-dried and ground to less than 45 mesh.

SAMPLE PREPARATION:Partial Extn.-Yes Total Extn.- % Extracted-45±15
Procedure- Weigh 0.500g sample into a calibrated digestion tube and add 1.5 ml HNO_3 . Mix and stand at room temperature for 2 hrs. Add 4.5 ml HCl to each tube, mix and stand at room temperature for another hour. Digest in a hot block for 1 hour at 40°C, 1 hour at 60°C, 1 hour at 80°C and 4 hrs at 100°C allowing 1/2 hr. ramping times
NOTE: The final volume should be less than 2 ml; if more, continue heating at 125°C to accomplish this.
Dilute with DDW to 25 ml and mix thoroughly.
Settle out suspended particles overnight and decant clear supernatant into disposable plastic tubes for analysis by AES-ICP. Samples requiring 5 or more metals are analyzed by AES-ICP (see below).
INTERFERENCES: Several, which are compensated for by the computer program.
REPORTING RESULTS: Two significant figures. µg/g Ca
INSTRUMENTATION: For the determination of 5 or more elements:
Inductively coupled Plasma Emission Spectrometer- Jobin-Yvon 1 m.JY48P with Autosampler & DEC computer.PET interface to LIS.(See "Remarks").
Calibration Range: 0 -1500 mg/l.(JA)
Resolution: Four significant figures (0.0001 mg/l).
Sensitivity: Not applicable (NA).
Instrument Detection Limit:0.2 mg/l.(JY)
PERFORMANCE CHARACTERISTICS:
Routine Operating Range-10 to 35000 µg/g.
Accuracy- Not determined (ND).
Precision of Controls-

	A	B
mean	28000µg/g	
std. dev.	2300 µg/g	
R.S.D.	8.4 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	230	480	760
mean	4500	11000	28000

W 200 µg/g T 1000 µg/g

CONTROL LIMITS: µg/g

Control		W.L. (x ±2σ)	R.L. (x ±3σ)
QCRS85-1	L.L.	23000	21000
	U.L.	33000	35000

REMARKS:- Determination of boron MUST be on Atom Scan 2400 only.
- Extraction % (±) based on average recovery of 4 samples from Soil Research Institute - SO1, SO2, SO3 & SO4.
- Samples requiring only Mg, Al and/or Ca are analyzed by Atom Scan.

SUMMARY REPORT OF QUALITY CONTROL DATA

CALCIUM IN SEDIMENT

Operating Range = 10.000to 35000. ug/g

IN - RUN DUPLICATES

Range	<10.000	10.000to7000.0	7000.0to17500.	17500.to35000.	>35000.
no.	7	12	9	1	15
s.w.		227.6760	478.5740	763.6750	
mean		4499.490	10545.76	27282.35	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
QCRS85-1	118	27661.94	2325.194	8.41

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
rb	25	6.250	7.8500

DATE 07/04/88

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: CALCIUM TEST CODE: CAUT SAMPLE TYPE: SEDIMENT
UNIT: Vegetation/Soil/Sediment SUPERVISOR: L. Pastorek

METHOD CODE: HMARSOIL - ICP/AES
REVISION NO: Original DATE: 1980
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- 10 to 20 g of wet sample
Container- 500 ml PET jar.
Preservative- None
Other- Samples are air-dried and ground to less than 45 mesh.

SAMPLE PREPARATION:Partial Extn.-Yes Total Extn.- % Extracted-45±15
Procedure- Weigh 0.500g sample into a calibrated digestion tube and add 1.5 ml HNO3 . Mix and stand at room temperature for 2 hrs. Add 4.5 ml HCl to each tube, mix and stand at room temperature for another hour. Digest in a hot block for 1 hour at 40°C, 1 hour at 60°C, 1 hour at 80°C and 4 hrs at 100°C allowing 1/2 hr. ramping times
NOTE: The final volume should be less than 2 ml; if more, continue heating at 125°C to accomplish this.
Dilute with DDW to 25 ml and mix thoroughly.
Settle out suspended particles overnight and decant clear supernatant into disposable plastic tubes for analysis by AES-ICP. Samples requiring 5 or more metals are analyzed by AES-ICP (see below).
INTERFERENCES: Several, which are compensated for by the computer program.
REPORTING RESULTS: Two significant figures. µg/g Ca
INSTRUMENTATION: For the determination of less than 5 elements: Inductively coupled Plasma Emission Spectrometer- Atomscan 2400 with Autosampler.(See "Remarks").
Calibration Range: 0 -2000 mg/l (AS-2400).
Resolution: Four significant figures (0.0001 mg/l).
Sensitivity: Not applicable (NA).
Instrument Detection Limit:4.0 mg/l (AS-2400).
PERFORMANCE CHARACTERISTICS:
Routine Operating Range-AS2400 200 -25000 µg/g
Accuracy- Not determined (ND).
Precision of Controls- A B
mean
std. dev.
R.S.D. %
Precision of Duplicates-low range mid range high range
s.d.
mean
W 0.2 µg/g T 1.0 µg/g

CONTROL LIMITS: µg/g		W.L. (x ±2σ)	R.L. (x ±3σ)
Control	L.L.	23000	21000
QCRS85-1	U.L.	33000	35000

REMARKS:- Determination of boron MUST be on Atom Scan 2400 only.
- Extraction % (±) based on average recovery of 4 samples from Soil Research Institute - SO1, SO2, SO3 & SO4.
- Samples requiring only Mg, Al and/or Ca are analyzed by Atom Scan.

SUMMARY REPORT OF QUALITY CONTROL DATA

CALCIUM-jy IN SEDIMENT

Operating Range = 20.000to 35000. ug/g

IN - RUN DUPLICATES

Range	<20.000	20.000to7000.0	7000.0to17500.	17500.to35000.	>35000.
no.	7	12	9	1	15
s.w.		227.6760	478.5740	763.6750	
mean		4499.490	10545.76	27282.35	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
QCRS85-1	118	27661.94	2325.194	8.41

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
rb	25	6.250	7.8500

DATE 87/04/22

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: CADMIUM TEST CODE: CDUT SAMPLE TYPE: SEDIMENT
UNIT: Vegetation/Soil/Sediment SUPERVISOR: L. Pastorek

METHOD CODE:535AA0 HMARSOIL - ICP/AES
REVISION NO: Original DATE: 1980
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- 10 to 20 g of wet sample
Container- 500 ml PET jar
Preservative- None
Other- Samples are air-dried and ground to less than 45 mesh.

SAMPLE PREPARATION:Partial Extn.-Yes Total Extn.- % Extracted-79±9
Procedure- Weigh 0.500g sample into a calibrated digestion tube and add 1.5 ml HNO_3 . Mix and stand at room temperature for 2 hrs. Add 4.5 ml HCl to each tube, mix and stand at room temperature for another hour. Digest in a hot block for 1 hour at 40°C, 1 hour at 60°C, 1 hour at 80°C and 4 hrs at 100°C allowing 1/2 hr. ramping times
NOTE: The final volume should be less than 2 ml; if more, continue heating at 125°C to accomplish this.
Dilute with DDW to 25 ml and mix thoroughly.
Settle out suspended particles overnight and decant clear supernatant into disposable plastic tubes for analysis by AES-ICP. Samples requiring 5 or more metals are analyzed by AES-ICP (see below).
INTERFERENCES: Several, which are compensated for by the computer program.
REPORTING RESULTS: Two significant figures. µg/g Cd
INSTRUMENTATION: For the determination of 5 or more elements:
Inductively coupled Plasma Emission Spectrometer- Jobin-Yvon 1 m.JY48P with Autosampler & DEC computer.PET interface to LIS.(See "Remarks").
Calibration Range: 0 - 1 mg/l.
Resolution: Four significant figures (0.0001 mg/l).
Sensitivity: Not applicable (NA).
Instrument Detection Limit: .002 mg/l.
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.1 to 5 µg/g.
Accuracy- Not determined (ND).
Precision of Controls-

	A	B
mean	12 µg/g	
std. dev.	0.81 µg/g	
R.S.D.	6.5 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	0.069	0.11	0.17
mean	0.54	1.8	3.3

W 0.05 µg/g T 0.25 µg/g

CONTROL LIMITS: µg/g		W.L. (x ±2σ)	R.L. (x ±3σ)
Control	L.L.	10	9.6
QCRS85-1	U.L.	14	14

REMARKS:- Determination of boron MUST be on Atom Scan 2400 only.
- Extraction % (±) based on average recovery of 4 samples from Soil Research Institute - SO1, SO2, SO3 & SO4.
- Samples requiring only Mg, Al and/or Ca are analyzed by Atom Scan.

SUMMARY REPORT OF QUALITY CONTROL DATA

CADMIUM IN SEDIMENT

Operating Range = 0.100 to 5.0 ug/g

IN - RUN DUPLICATES

Range	<0.100	0.100 to1.00	1.00 to2.50	2.50 to5.0	>5.0
no.	19	13	6	3	3
s.w.		0.0805	0.1059	0.1678	
mean		0.5074	1.8012	3.3287	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
QCRS85-1	121	12.457	0.8090	6.49

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
rb	30	0.045	0.0665

DATE 87/04/23

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: COBALT TEST CODE: COUT SAMPLE TYPE: SEDIMENT
UNIT: Vegetation/Soil/Sediment SUPERVISOR: L. Pastorek

METHOD CODE:535AA0 HMARSOIL - ICP/AES
REVISION NO: Original DATE: 1980
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- 10 to 20 g of wet sample
Container- 500 ml PET jar
Preservative- None
Other- Samples are air-dried and ground to less than 45 mesh.

SAMPLE PREPARATION:Partial Extn.-Yes Total Extn.- % Extracted-43±15
Procedure- Weigh 0.500g sample into a calibrated digestion tube and add 1.5 ml HNO3 . Mix and stand at room temperature for 2 hrs. Add 4.5 ml HCl to each tube, mix and stand at room temperature for another hour. Digest in a hot block for 1 hour at 40°C, 1 hour at 60°C, 1 hour at 80°C and 4 hrs at 100°C allowing 1/2 hr. ramping times
NOTE: The final volume should be less than 2 ml; if more, continue heating at 125°C to accomplish this.
Dilute with DDW to 25 ml and mix thoroughly.
Settle out suspended particles overnight and decant clear supernatant into disposable plastic tubes for analysis by AES-ICP. Samples requiring 5 or more metals are analyzed by AES-ICP (see below).
INTERFERENCES: Several, which are compensated for by the computer program.
REPORTING RESULTS: Two significant figures. µg/g Co
INSTRUMENTATION: For the determination of 5 or more elements:
Inductively coupled Plasma Emission Spectrometer- Jobin-Yvon 1 m.JY48P with Autosampler & DEC computer.PET interface to LIS.(See "Remarks").
Calibration Range: 0 - 10 mg/l.
Resolution: Four significant figures (0.0001 mg/l).
Sensitivity: Not applicable (NA).
Instrument Detection Limit: .003 mg/l.
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.2 to 50 µg/g.
Accuracy- Not determined (ND).
Precision of Controls-

	A	B
mean	36 µg/g	
std. dev.	3.3 µg/g	
R.S.D.	9.2 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	0.37	1.1	ND
mean	6.4	15	ND

W 0.2 µg/g T 1.0 µg/g

CONTROL LIMITS: µg/g

		W.L. (x ±2σ)	R.L. (x ±3σ)
Control	L.L.	29	26
QCRS85-1	U.L.	43	46

REMARKS:- Determination of boron MUST be on Atom Scan 2400 only.
- Extraction % (±) based on average recovery of 4 samples from Soil Research Institute - SO1, SO2, SO3 & SO4.
- Samples requiring only Mg, Al and/or Ca are analyzed by Atom Scan.

SUMMARY REPORT OF QUALITY CONTROL DATA

COBALT IN SEDIMENT

Operating Range = 0.200 to 50.0 ug/g

IN - RUN DUPLICATES

Range	<0.200	0.200 to10.00	10.00 to25.00	25.00 to50.0	>50.0
no.	3	18	23	0	0
s.w.		0.3748	1.0719	0.0000	
mean		6.3745	14.6192	0.0000	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
QCRS85-1	119	35.626	3.2747	9.19

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
rb	45	0.135	0.1725

DATE 87/04/23

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: CHROMIUM TEST CODE: CRUT SAMPLE TYPE: SEDIMENT
UNIT: Vegetation/Soil/Sediment SUPERVISOR: L. Pastorek

METHOD CODE:535AA0 HMARSOIL - ICP/AES
REVISION NO: Original DATE: 1980
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- 10 to 20 g of wet sample
Container- 500 ml PET jar
Preservative- None
Other- Samples are air-dried and ground to less than 45 mesh.

SAMPLE PREPARATION:Partial Extn.-Yes Total Extn.- % Extracted-56±20
Procedure- Weigh 0.500g sample into a calibrated digestion tube and add 1.5 ml HNO3 . Mix and stand at room temperature for 2 hrs. Add 4.5 ml HCl to each tube, mix and stand at room temperature for another hour. Digest in a hot block for 1 hour at 40°C, 1 hour at 60°C, 1 hour at 80°C and 4 hrs at 100°C allowing 1/2 hr. ramping times
NOTE: The final volume should be less than 2 ml; if more, continue heating at 125°C to accomplish this.
Dilute with DDW to 25 ml and mix thoroughly.
Settle out suspended particles overnight and decant clear supernatant into disposable plastic tubes for analysis by AES-ICP. Samples requiring 5 or more metals are analyzed by AES-ICP (see below).
INTERFERENCES: Several, which are compensated for by the computer program.
REPORTING RESULTS: Two significant figures. µg/g Cr
INSTRUMENTATION: For the determination of 5 or more elements:
Inductively coupled Plasma Emission Spectrometer- Jobin-Yvon 1 m.JY48P with Autosampler & DEC computer.PET interface to LIS.(See "Remarks").
Calibration Range: 0 - 10 mg/l.
Resolution: Four significant figures (0.0001 mg/l).
Sensitivity: Not applicable (NA).
Instrument Detection Limit:.007 mg/l.
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.35 to 100 µg/g.
Accuracy- Not determined (ND).
Precision of Controls-

	A	B
mean	220 µg/g	
std. dev.	26 µg/g	
R.S.D.	12 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	1.5	1.4	2.6
mean	14	33	62

W 1 µg/g T 5 µg/g

CONTROL LIMITS: µg/g

		W.L. (x ±2σ)	R.L. (x ±3σ)
Control	L.L.	170	140
QCRS85-1	U.L.	270	300

REMARKS:- Determination of boron MUST be on Atom Scan 2400 only.
- Extraction % (±) based on average recovery of 4 samples from Soil Research Institute - SO1, SO2, SO3 & SO4.
- Samples requiring only Mg, Al and/or Ca are analyzed by Atom Scan.

SUMMARY REPORT OF QUALITY CONTROL DATA

CHROMIUM IN SEDIMENT

Operating Range = 0.350 to 100.0 ug/g

IN - RUN DUPLICATES

Range	<0.350	0.350 to20.00	20.00 to50.00	50.00 to100.0	>100.0
no.	4	8	17	9	6
s.w.		1.4886	1.4513	2.5668	
mean		13.9045	33.3531	62.3398	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
QCRS85-1	121	219.433	25.5221	11.63

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
rb	79	1.605	2.6355

DATE 87/04/23

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: COPPER TEST CODE: CUUT SAMPLE TYPE: SEDIMENT
UNIT: Vegetation/Soil/Sediment SUPERVISOR: L. Pastorek

METHOD CODE:535AA0 HMARSOIL - ICP/AES
REVISION NO: Original DATE: 1980
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required-10 to 20g of wet sample
Container- 500 ml PET jar
Preservative- None
Other- Samples are air-dried and ground to less than 45 mesh.

SAMPLE PREPARATION:Partial Extn.-Yes Total Extn.- % Extracted-70±7
Procedure- Weigh 0.500g sample into a calibrated digestion tube and add 1.5 ml HNO3 . Mix and stand at room temperature for 2 hrs. Add 4.5 ml HCl to each tube, mix and stand at room temperature for another hour. Digest in a hot block for 1 hour at 40°C, 1 hour at 60°C, 1 hour at 80°C and 4 hrs at 100°C allowing 1/2 hr. ramping times
NOTE: The final volume should be less than 2 ml; if more, continue heating at 125°C to accomplish this.
Dilute with DDW to 25 ml and mix thoroughly.
Settle out suspended particles overnight and decant clear supernatant into disposable plastic tubes for analysis by AES-ICP. Samples requiring 5 or more metals are analyzed by AES-ICP (see below).
INTERFERENCES: Several, which are compensated for by the computer program.
REPORTING RESULTS: Two significant figures. (µg/g Cu)
INSTRUMENTATION: For the determination of 5 or more elements:
Inductively coupled Plasma Emission Spectrometer- Jobin-Yvon 1 m.JY48P with Autosampler & DEC computer.PET interface to LIS.(See "Remarks").
Calibration Range: 0 - 10 mg/l.
Resolution: Four significant figures (0.0001 mg/l).
Sensitivity: Not applicable (NA).
Instrument Detection Limit: .006 mg/l.
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.3 to 100 µg/g.
Accuracy- Not determined (ND).
Precision of Controls-

	A	B
mean	1600 µg/g	
std. dev.	150 µg/g	
R.S.D.	9.7 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	1.1	1.5	3.2
mean	11	33	81

W 1 µg/g T 5 µg/g

CONTROL LIMITS: µg/g

Control		W.L. (x ±2σ)	R.L. (x ±3σ)
QCRS85-1	L.L.	1300	1200
	U.L.	1900	2000

REMARKS:- Determination of boron MUST be on Atom Scan 2400 only.
- Extraction % (±) based on average recovery of 4 samples from Soil Research Institute - SO1, SO2, SO3 & SO4.
- Samples requiring only Mg, Al and/or Ca are analyzed by Atom Scan.

SUMMARY REPORT OF QUALITY CONTROL DATA

COPPER IN SEDIMENT

Operating Range = 0.300 to 100.0 ug/g

IN - RUN DUPLICATES

Range	<0.300	0.300 to20.00	20.00 to50.00	50.00 to100.0	>100.0
no.	1	12	19	3	9
s.w.		1.0539	1.5028	3.2269	
mean		11.1881	32.5925	81.2098	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
QCRS85-1	121	1554.756	150.3681	9.67

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
rb	71	1.255	3.0840

DATE 87/04/23

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: IRON TEST CODE: FEUT SAMPLE TYPE: SEDIMENT
UNIT: Vegetation/Soil/Sediment SUPERVISOR: L. Pastorek

METHOD CODE:535AA0 HMARSOIL - ICP/AES
REVISION NO: Original DATE: 1980
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- 10 to 20 g of wet sample
Container- 500 ml PET jar
Preservative- None
Other- Samples are air-dried and ground to less than 45 mesh.

SAMPLE PREPARATION:Partial Extn.-Yes Total Extn.- % Extracted-66±13
Procedure- Weigh 0.500g sample into a calibrated digestion tube and add 1.5 ml HNO3 . Mix and stand at room temperature for 2 hrs. Add 4.5 ml HCl to each tube, mix and stand at room temperature for another hour. Digest in a hot block for 1 hour at 40°C, 1 hour at 60°C, 1 hour at 80°C and 4 hrs at 100°C allowing 1/2 hr. ramping times
NOTE: The final volume should be less than 2 ml; if more, continue heating at 125°C to accomplish this.
Dilute with DDW to 25 ml and mix thoroughly.
Settle out suspended particles overnight and decant clear supernatant into disposable plastic tubes for analysis by AES-ICP. Samples requiring 5 or more metals are analyzed by AES-ICP (see below).
INTERFERENCES: Several, which are compensated for by the computer program.
REPORTING RESULTS: Two significant figures. µg/g Fe
INSTRUMENTATION: For the determination of 5 or more elements:
Inductively coupled Plasma Emission Spectrometer- Jobin-Yvon 1 m.JY48P with Autosampler & DEC computer.PET interface to LIS.(See "Remarks").
Calibration Range: 0 - 1000 mg/l.
Resolution: Four significant figures (0.0001 mg/l).
Sensitivity: Not applicable (NA).
Instrument Detection Limit:.02-2 mg/l.
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 1.0 to 50000 µg/g.
Accuracy- Not determined (ND).
Precision of Controls-

	A	B
mean	27000µg/g	
std. dev.	1300 µg/g	
R.S.D.	4.7 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	440	570	1900
mean	7000	1800	34000

W .2 µg/g T 1.0 µg/g

CONTROL LIMITS: µg/g

		W.L. (x ±2σ)	R.L. (x ±3σ)
Control	L.L.	24000	23000
QCRS85-1	U.L.	30000	31000

REMARKS:- Determination of boron MUST be on Atom Scan 2400 only.
- Extraction % (±) based on average recovery of 4 samples from Soil Research Institute - SO1, SO2, SO3 & SO4.
- Samples requiring only Mg, Al and/or Ca are analyzed by Atom Scan.

SUMMARY REPORT OF QUALITY CONTROL DATA

IRON IN SEDIMENT

Operating Range = 1.000 to 50000. ug/g

IN - RUN DUPLICATES

Range	<1.000	1.000 to10000.	10000.to25000.	25000.to50000.	>50000.
no.	1	10	18	13	2
s.w.		437.0540	570.8940	1856.594	
mean		7025.080	17742.55	33587.18	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
QCRS85-1	122	26811.70	1261.599	4.71

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
rb	30	1.800	6.1250

DATE 87/04/22

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: LEAD TEST CODE: PBUT SAMPLE TYPE: SEDIMENT
UNIT: Vegetation/Soil/Sediment SUPERVISOR: L. Pastorek

METHOD CODE:535AA0 HMARSOIL - ICP/AES
REVISION NO: Original DATE: 1980
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- 10 to 20 g of wet sample
Container- 500 ml PET jar
Preservative- None
Other- Samples are air-dried and ground to less than 45 mesh.

SAMPLE PREPARATION:Partial Extn.-Yes Total Extn.- % Extracted-85±15
Procedure- Weigh 0.500g sample into a calibrated digestion tube and add 1.5 ml HNO_3 . Mix and stand at room temperature for 2 hrs. Add 4.5 ml HCl to each tube, mix and stand at room temperature for another hour. Digest in a hot block for 1 hour at 40°C, 1 hour at 60°C, 1 hour at 80°C and 4 hrs at 100°C allowing 1/2 hr. ramping times
NOTE: The final volume should be less than 2 ml; if more, continue heating at 125°C to accomplish this.
Dilute with DDW to 25 ml and mix thoroughly.
Settle out suspended particles overnight and decant clear supernatant into disposable plastic tubes for analysis by AES-ICP. Samples requiring 5 or more metals are analyzed by AES-ICP (see below).
INTERFERENCES: Several, which are compensated for by the computer program.
REPORTING RESULTS: Two significant figures. µg/g Pb
INSTRUMENTATION: For the determination of 5 or more elements:
Inductively coupled Plasma Emission Spectrometer- Jobin-Yvon 1 m.JY48P with Autosampler & DEC computer.PET interface to LIS.(See "Remarks").
Calibration Range: 0 - 10 mg/l.
Resolution: Four significant figures (0.0001 mg/l).
Sensitivity: Not applicable (NA).
Instrument Detection Limit: .02 mg/l.
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 1.0 to 200 µg/g.
Accuracy- Not determined (ND).
Precision of Controls-

	A	B
mean	940 µg/g	
std. dev.	94 µg/g	
R.S.D.	10 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	1.2	3.7	14
mean	16	65	140

W .5 µg/g T 2.5 µg/g

CONTROL LIMITS: µg/g

Control		W.L. (x ±2σ)	R.L. (x ±3σ)
QCRS85-1	L.L.	750	660
	U.L.	1100	1200

REMARKS:- Determination of boron MUST be on Atom Scan 2400 only.
- Extraction % (±) based on average recovery of 4 samples from Soil Research Institute - SO1, SO2, SO3 & SO4.
- Samples requiring only Mg, Al and/or Ca are analyzed by Atom Scan.

SUMMARY REPORT OF QUALITY CONTROL DATA

LEAD IN SEDIMENT

Operating Range = 1.000 to 200.0 ug/g

IN - RUN DUPLICATES

Range	<1.000	1.000 to40.00	40.00 to100.00	100.00to200.0	>200.0
no.	2	25	7	5	5
s.w.		1.2491	3.6861	14.4014	
mean		15.9352	65.0354	143.6799	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
QCRS85-1	122	940.243	94.4859	10.05

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
rb	62	1.330	0.8880

DATE 87/04/23

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: MAGNESIUM TEST CODE: MGUT SAMPLE TYPE: SEDIMENT
UNIT: Vegetation/Soil/Sediment SUPERVISOR: L. Pastorek

METHOD CODE:535AA0 HMARSOIL - ICP/AES
REVISION NO: Original DATE: 1980
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- 10 to 20 g of wet sample
Container- 500 ml PET jar
Preservative- None
Other- Samples are air-dried and ground to less than 45 mesh.

SAMPLE PREPARATION:Partial Extn.-Yes Total Extn.- % Extracted-86±25
Procedure- Weigh 0.500g sample into a calibrated digestion tube and add 1.5 ml HNO3 . Mix and stand at room temperature for 2 hrs. Add 4.5 ml HCl to each tube, mix and stand at room temperature for another hour. Digest in a hot block for 1 hour at 40°C, 1 hour at 60°C, 1 hour at 80°C and 4 hrs at 100°C allowing 1/2 hr. ramping times
NOTE: The final volume should be less than 2 ml; if more, continue heating at 125°C to accomplish this.
Dilute with DDW to 25 ml and mix thoroughly.
Settle out suspended particles overnight and decant clear supernatant into disposable plastic tubes for analysis by AES-ICP. Samples requiring 5 or more metals are analyzed by AES-ICP (see below).
INTERFERENCES: Several, which are compensated for by the computer program.
REPORTING RESULTS: Two significant figures. µg/g Mg
INSTRUMENTATION: For the determination of 5 or more elements:
Inductively coupled Plasma Emission Spectrometer- Jobin-Yvon 1 m.JY48P with Autosampler & DEC computer.PET interface to LIS.(See "Remarks").
Calibration Range: 0 -50 mg/l.(
Resolution: Four significant figures (0.0001 mg/l).
Sensitivity: Not applicable (NA).
Instrument Detection Limit:0.2 mg/l
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 10 to 20000 µg/g.
Accuracy- Not determined (ND).
Precision of Controls-

	A	B
mean	5600 µg/g	
std. dev.	280 µg/g	
R.S.D.	5.1 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	140	230	820
mean	2400	7200	13000

W 100 µg/g T 500 µg/g

CONTROL LIMITS: µg/g

Control		W.L. (x ±2σ)	R.L. (x ±3σ)
QCRS85-1	L.L.	5000	4800
	U.L.	6200	6400

REMARKS:- Determination of boron MUST be on Atom Scan 2400 only.
- Extraction % (±) based on average recovery of 4 samples from Soil Research Institute - SO1, SO2, SO3 & SO4.
- Samples requiring only Mg, Al and/or Ca are analyzed by Atom Scan.

SUMMARY REPORT OF QUALITY CONTROL DATA

MAGNESIUM IN SEDIMENT

Operating Range = 10.000to 20000. ug/g

IN - RUN DUPLICATES

Range	<10.000	10.000to4000.0	4000.0to10000.	10000.to20000.	>20000.
no.	1	11	15	15	2
s.w.		140.1940	231.9700	817.1540	
mean		2422.710	7197.330	12879.04	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
QCRS85-1	122	5555.050	282.4490	5.08

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
rb	20	2.500	2.3900

DATE 87/04/22

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: MANGANESE TEST CODE: MNUT SAMPLE TYPE: SEDIMENT
UNIT: Vegetation/Soil/Sediment SUPERVISOR: L. Pastorek

METHOD CODE:535AA0 HMARSOIL - ICP/AES
REVISION NO: Original DATE: 1980
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- 10 to 20 g of wet sample
Container- 500 ml PET jar
Preservative- None
Other- Samples are air-dried and ground to less than 45 mesh.

SAMPLE PREPARATION:Partial Extn.-Yes Total Extn.- % Extracted-69±15
Procedure- Weigh 0.500g sample into a calibrated digestion tube and add 1.5 ml HNO3 . Mix and stand at room temperature for 2 hrs. Add 4.5 ml HCl to each tube, mix and stand at room temperature for another hour. Digest in a hot block for 1 hour at 40°C, 1 hour at 60°C, 1 hour at 80°C and 4 hrs at 100°C allowing 1/2 hr. ramping times
NOTE: The final volume should be less than 2 ml; if more, continue heating at 125°C to accomplish this.
Dilute with DDW to 25 ml and mix thoroughly.
Settle out suspended particles overnight and decant clear supernatant into disposable plastic tubes for analysis by AES-ICP. Samples requiring 5 or more metals are analyzed by AES-ICP (see below).
INTERFERENCES: Several, which are compensated for by the computer program.
REPORTING RESULTS: Two significant figures. µg/g Mn
INSTRUMENTATION: For the determination of 5 or more elements:
Inductively coupled Plasma Emission Spectrometer- Jobin-Yvon 1 m.JY48P with Autosampler & DEC computer.PET interface to LIS.(See "Remarks").
Calibration Range: 0 - 10 mg/l.
Resolution: Four significant figures (0.0001 mg/l).
Sensitivity: Not applicable (NA).
Instrument Detection Limit:.008 mg/l.
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.4 to 1000 µg/g.
Accuracy- Not determined (ND).
Precision of Controls-

	A	B
mean	390 µg/g	
std. dev.	38 µg/g	
R.S.D.	9.7 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	7.4	15	14
mean	130	330	620

W .005 µg/g T .025 µg/g

CONTROL LIMITS: µg/g

Control		W.L. (x ±2σ)	R.L. (x ±3σ)
QCRS85-1	L.L.	310	280
	U.L.	470	500

REMARKS:- Determination of boron MUST be on Atom Scan 2400 only.
- Extraction % (±) based on average recovery of 4 samples from Soil Research Institute - SO1, SO2, SO3 & SO4.
- Samples requiring only Mg, Al and/or Ca are analyzed by Atom Scan.

SUMMARY REPORT OF QUALITY CONTROL DATA

MANGANESE IN SEDIMENT

Operating Range = 0.400 to 1000.0 ug/g

IN - RUN DUPLICATES

Range	<0.400	0.400 to200.00	200.00to500.00	500.00to1000.0	>1000.0
no.	2	9	21	10	2
s.w.		7.3870	15.1967	13.8967	
mean		130.2543	332.1617	617.9534	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
QCRS85-1	122	393.609	38.0969	9.68

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
rb	32	0.140	0.0975

DATE 87/04/02

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME:Mercury TEST CODE:HGUT SAMPLE TYPE:SEDIMENT
UNIT:Biomaterials SUPERVISOR: R. S. Sadana

METHOD CODE:541AF1 TYPE: Flameless AAS
REVISION NO: Original DATE: May, 1984
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- 10 to 20 g wet sample
Container- 500 ml PET jar
Preservative- None
Other- Samples are air-dried and ground to less than 45 mesh

SAMPLE PREPARATION:Partial Extn.- Total Extn.-Yes % Extracted-
Procedure- Weigh approx. 0.250 g of sample into a 50 ml Folin-Wu digestion tube. Add 5 ml of acid mixture (4:1 -H2SO4:HNO3) and place the tube in a Technicon aluminum hot block (1½h @ 150°C;1½h @ 190°C;2h @ 250°C).
Cool overnight,then dilute to 25 ml with distilled water.
Run in batches of about 35 samples.

Treat blanks and calibration standards in exactly the same manner. Determine mercury in the entire volume. The measurement step is automated and is based on the evolution of atomic vapour of mercury (wavelength - 254nm) by the addition of a reducing agent.
INTERFERENCES: Water vapour; organic solvents.
Very high concentration of cations.
REPORTING RESULTS:Two significant figures (ug/g).
INSTRUMENTATION: Automated sampler and peristaltic pump (Technicon or Gilson). Laboratory Data Control U.V. monitor (Pharmacia or Milton -Roy).
Calibration Range: 0 - 20.0 ng/ml
Resolution: 0.4 ng/ml (one division on recorder chart paper)
Sensitivity:1.0 ng/ml reads 0.05 absorbance (2.5 divs on chart)
Instrument Detection Limit: 0.1 ng/ml
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.01 - 2.0 µg/g
Accuracy- 90% at 1.1 ug/g
Precision of Controls-

	A	B
mean	.45 µg/g	
std. dev.	.035 µg/g	
R.S.D.	7.6 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	.010	.059	.071
mean	.096	.45	1.3

W 0.01 µg/g T 0.05 µg/g

CONTROL LIMITS:

REMARKS:

- Precision based on CCIW round robin.
- Detection Limit - 2x std. dev. of low range within-run duplicates.
- Accuracy - Ratio of mean to cert. value in ref. mat. (%).

SUMMARY REPORT OF QUALITY CONTROL DATA

MERCURY IN SEDIMENT

Operating Range = 0.010 to 2.0 ug/g

IN - RUN DUPLICATES

Range	<0.010	0.010 to0.40	0.40 to1.00	1.00 to2.0	>2.0
no.	2	18	2	1	1
s.w.		0.0102	0.0591	0.0707	
mean		0.0960	0.4520	1.3300	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
con 684	104	0.454	0.0347	7.64

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
blk	0	0	0

DATE 86/03/31

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME:MOLYBDENUM TEST CODE: MOUT SAMPLE TYPE: SEDIMENT
UNIT: Vegetation/Soil/Sediment SUPERVISOR: L. Pastorek

METHOD CODE:535AA0 HMARSOIL - ICP/AES
REVISION NO: Original DATE: 1980
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- 10 to 20 g of wet sample
Container- 500 ml PET jar
Preservative- None
Other- Samples are air-dried and ground to less than 45 mesh.

SAMPLE PREPARATION:Partial Extn.-Yes Total Extn.- % Extracted-ND
Procedure- Weigh 0.500g sample into a calibrated digestion tube and add 1.5 ml HNO_3 . Mix and stand at room temperature for 2 hrs. Add 4.5 ml HCl to each tube, mix and stand at room temperature for another hour. Digest in a hot block for 1 hour at 40°C, 1 hour at 60°C, 1 hour at 80°C and 4 hrs at 100°C allowing 1/2 hr. ramping times
NOTE: The final volume should be less than 2 ml; if more, continue heating at 125°C to accomplish this.
Dilute with DDW to 25 ml and mix thoroughly.
Settle out suspended particles overnight and decant clear supernatant into disposable plastic tubes for analysis by AES-ICP. Samples requiring 5 or more metals are analyzed by AES-ICP (see below).
INTERFERENCES: Several, which are compensated for by the computer program.
REPORTING RESULTS: Two significant figures. µg/g Mo
INSTRUMENTATION: For the determination of 5 or more elements:
Inductively coupled Plasma Emission Spectrometer- Jobin-Yvon 1 m.JY48P with Autosampler & DEC computer.PET interface to LIS.(See "Remarks").
Calibration Range: 0 - 10 mg/l.
Resolution: Four significant figures (0.0001 mg/l).
Sensitivity: Not applicable (NA).
Instrument Detection Limit:.004 mg/l.
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.2 to 10 µg/g.
Accuracy- Not determined (ND).
Precision of Controls-

	A	B
mean	6.1 µg/g	
std. dev.	0.87 µg/g	
R.S.D.	14 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	0.18	0.24	0.92
mean	1.5	3.1	6.6

W 0.5 µg/g T 2.5 µg/g

CONTROL LIMITS: µg/g

Control		W.L. (x ±2σ)	R.L. (x ±3σ)
QCRS85-1	L.L.	4.4	3.5
	U.L.	7.8	8.7

REMARKS:- Determination of boron MUST be on Atom Scan 2400 only.
- Extraction % (±) based on average recovery of 4 samples from Soil Research Institute - SO1, SO2, SO3 & SO4.
- Samples requiring only Mg, Al and/or Ca are analyzed by Atom Scan.

SUMMARY REPORT OF QUALITY CONTROL DATA

MOLYBDENUM IN SEDIMENT

Operating Range = 0.200 to 10.0 ug/g

IN - RUN DUPLICATES

Range	<0.200	0.200 to2.00	2.00 to5.00	5.00 to10.0	>10.0
no.	33	3	6	2	0
s.w.		0.5193	0.2359	0.9253	
mean		1.2196	3.1184	6.5644	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
QCRS85-1	120	6.114	0.8722	14.27

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
rb	54	0.365	0.2775

DATE 87/04/23

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: NICKEL TEST CODE: NIUT SAMPLE TYPE: SEDIMENT
UNIT: Vegetation/Soil/Sediment SUPERVISOR: L. Pastorek

METHOD CODE:535AA0 HMARSOIL - ICP/AES
REVISION NO: Original DATE: 1980
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- 10 to 20g of wet sample
Container- 500 ml PET jar
Preservative- None
Other- Samples are air-dried and ground to less than 45 mesh.

SAMPLE PREPARATION:Partial Extn.-Yes Total Extn.- % Extracted-69±7
Procedure- Weigh 0.500g sample into a calibrated digestion tube and add 1.5 ml HNO_3 . Mix and stand at room temperature for 2 hrs. Add 4.5 ml HCl to each tube, mix and stand at room temperature for another hour. Digest in a hot block for 1 hour at 40°C, 1 hour at 60°C, 1 hour at 80°C and 4 hrs at 100°C allowing 1/2 hr. ramping times
NOTE: The final volume should be less than 2 ml; if more, continue heating at 125°C to accomplish this.
Dilute with DDW to 25 ml and mix thoroughly.
Settle out suspended particles overnight and decant clear supernatant into disposable plastic tubes for analysis by AES-ICP. Samples requiring 5 or more metals are analyzed by AES-ICP (see below).
INTERFERENCES: Several, which are compensated for by the computer program.
REPORTING RESULTS: Two significant figures. µg/g Ni
INSTRUMENTATION: For the determination of 5 or more elements:
Inductively coupled Plasma Emission Spectrometer- Jobin-Yvon 1 m.JY48P with Autosampler & DEC computer.PET interface to LIS.(See "Remarks").
Calibration Range: 0 - 10 mg/l.
Resolution: Four significant figures (0.0001 mg/l).
Sensitivity: Not applicable (NA).
Instrument Detection Limit: .004 mg/l.
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.2 to 100 µg/g.
Accuracy- Not determined (ND).
Precision of Controls-

	A	B
mean	620 µg/g	
std. dev.	63 µg/g	
R.S.D.	10 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	0.51	1.1	4.6
mean	10	28	70

W 1 µg/g T 5 µg/g

CONTROL LIMITS: µg/g

Control		W.L. (x ±2σ)	R.L. (x ±3σ)
QCRS85-1	L.L.	490	430
	U.L.	750	810

REMARKS:- Determination of boron MUST be on Atom Scan 2400 only.
- Extraction % (±) based on average recovery of 4 samples from Soil Research Institute - SO1, SO2, SO3 & SO4.
- Samples requiring only Mg, Al and/or Ca are analyzed by Atom Scan.

SUMMARY REPORT OF QUALITY CONTROL DATA

NICKEL IN SEDIMENT

Operating Range = 0.200 to 100.0 ug/g

IN - RUN DUPLICATES

Range	<0.200	0.200 to20.00	20.00 to50.00	50.00 to100.0	>100.0
no.	1	21	15	4	3
s.w.		0.5111	1.1288	4.5547	
mean		10.0851	27.8792	69.6108	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
QCRS85-1	120	624.781	62.5520	10.01

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
rb	66	0.980	1.5095

DATE 87/04/23

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME:Selenium TEST CODE:SEUT SAMPLE TYPE:SEDIMENT
UNIT: Biomaterials SUPERVISOR: R. S. Sadana

METHOD CODE:510EF3 TYPE: Semi-aut. hydr. gen - flameless AAS
REVISION NO: Original DATE: January, 1983
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- 10 to 20 g of wet sample
Container- 500 ml PET jar
Preservative- None
Other- Samples are air-dried and gound to less than 45 mesh

SAMPLE PREPARATION:Partial Extn.- Total Extn.-Yes % Extracted->90
Procedure- Weigh 60 mg (45 mesh) sample into a 18 x 150 mm pyrex graduated test tube. Add 3 ml of acid mixture (6 nitric: 3 sulphuric: 1 perchloric). Process in batches of 80 samples including blanks, calibration standards and controls.
Digest in an aluminum hot block at a medium setting on the hot plate for 14 hrs until dense white fumes appear.
Cool,add 0.5 ml of distilled water, then add 2 ml conc. HCl.
Dilute to 15 ml with distilled water and mix.
Feed the prepared solutions to the automated system for the determination of selenium by the hydride-FAAS technique.
INTERFERENCES: Excessive concentrations of Cu, Fe and Ni may interfere.
REPORTING RESULTS:2 dec. <10, 1 dec. <100, 0 dec. if >100 µg/g
INSTRUMENTATION: Atomic absorption spectrophotometer (Varian Techtron 1200 & AA-5,with chart recorder,peristaltic pump and autosampler.
Open ended heated quartz "T" cell; gas-liquid separator
Calibration Range:0 - 40 ng/ml (linear<20ng/ml)
Resolution: 0.01 absorbance (unexpanded scale)
Sensitivity:0.02 µg/ml reads 0.20 abs.
Instrument Detection Limit: 0.001 µg/ml
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.3 to 10 µg/g
Accuracy- 99% (NBS orchard leaves)
Precision of Controls-

	A	B
mean	.51 µg/g	.70 µg/g
std. dev.	.15 µg/g	.24 µg/g
R.S.D.	30 %	34 %

Precision of Duplicates-

	low range	mid range	high range
s.d.	.097	.13	ND
mean	.38	3.5	ND

W 0.2 µg/g T 1.0 µg/g

CONTROL LIMITS:

REMARKS:

- Detection Limit - 3x std. dev. of low range within-run duplicates.
- Accuracy - Ratio of mean to cert. value in ref. mat. (%).

SUMMARY REPORT OF QUALITY CONTROL DATA

SELENIUM IN SEDIMENT

Operating Range = 0.300 to 10.0 ug/g

IN - RUN DUPLICATES

Range	<0.300	0.300 to2.00	2.00 to5.00	5.00 to10.0	>10.0
no.	4	5	1	0	0
s.w.		0.1229	0.1273	0.0000	
mean		0.4980	3.4500	0.0000	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
orch leave	31	0.082	0.0523	63.78
veg contro	49	3.741	0.4038	10.79
soil cont1	66	0.510	0.1506	29.53
soil cont2	68	0.699	0.2396	34.28

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLK	0	0.000	0.0000

DATE 87/03/17

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: SILVER TEST CODE: AGUT SAMPLE TYPE: SEDIMENT
UNIT: Vegetation/Soil/Sediment SUPERVISOR: L. Pastorek

METHOD CODE:535AA0 HMARSOIL - AAS
REVISION NO: Original DATE: 1980
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- 10 to 20 g wet sample
Container- 500 ml PET jar
Preservative- None
Other- Samples are air-dried and ground to less than 45 mesh.

SAMPLE PREPARATION:Partial Extn.-Yes Total Extn.- % Extracted-ND
Procedure- Weigh 0.500g sample into a calibrated digestion tube and add 1 ml H_2SO_4 (conc.). Add 2.5 ml HNO_3 (conc) and mix well.

Digest in a hot block for 1 hour at 60°C, and 4.5 hrs at 175°C (allowing 1 hr. ramping times).
NOTE: The final volume should be less than 2 ml; if more, continue heating at 175°C to accomplish this. Samples MUST fume during digestion. Cool, dilute with DDW to 25 ml and mix thoroughly. Settle out suspended particles overnight and decant clear supernatant into disposable plastic tubes for analysis by AAS. For the determination of < 5 elements and/or Na, K or Ag use the PE-5000.
INTERFERENCES: Several, which may be compensated for by background correction.
REPORTING RESULTS: Two significant figures. µg/g Ag
INSTRUMENTATION: PE 5000 atomic absorption spectrophotometer, PE Autosampler 50 and a PE Automatic Burner Controller, interfaced with Commodore Pet 4032, Tractor Printer 4022P & CBM 805C dual drive.
Calibration Range: 0 - 2.0 mg/l.
Resolution: 0.001 mg/l.
Sensitivity: conc. for absorbance of approx. 0.20 units (2.5 mg/l)
Instrument Detection Limit: 0.04 mg/l.
PERFORMANCE CHARACTERISTICS:
Routine Operating Range-2 to 100 µg/g.
Accuracy- Not determined (ND).
Precision of Controls-

	A	B
mean	ND	
std. dev.	ND	
R.S.D.	ND	

Precision of Duplicates-

	low range	mid range	high range
s.d.	ND	ND	ND
mean	ND	ND	ND

W T

CONTROL LIMITS: µg/g W.L. (x ±2σ) R.L. (x ±3σ)
L.L.
U.L.
REMARKS: Control QCRS85-1
Extraction % (±) ND -based on average recovery of 4 samples from Soil Research Institute - S01, S02, S03 & S04.

SUMMARY REPORT OF QUALITY CONTROL DATA

SILVER IN SEDIMENT

Operating Range = 2.000 to 100.0 ug/g

IN - RUN DUPLICATES

Range	<2.000	2.000 to20.00	20.00 to50.00	50.00 to100.0	>100.0
no.	36	7	1	0	0
s.w.		1.0464	6.6901	0.0000	
mean		5.5643	23.7527	0.0000	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
QCRS85-1	20	2.607	1.5992	61.34

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
rb	18	0.190	0.2065

DATE 87/04/23

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: STRONTIUM TEST CODE: SRUT SAMPLE TYPE: SEDIMENT
UNIT: Vegetation/Soil/Sediment SUPERVISOR: L. Pastorek

METHOD CODE:533AA0 HMARSOIL - ICP/AES
REVISION NO: Original DATE: 1980
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- 10 to 20 g of wet sample
Container- 500 ml PET jar
Preservative- None
Other- Samples are air-dried and ground to less than 45 mesh.

SAMPLE PREPARATION:Partial Extn.-Yes Total Extn.- % Extracted-ND
Procedure- Weigh 0.500g sample into a calibrated digestion tube and add 1.5 ml HNO3 . Mix and stand at room temperature for 2 hrs. Add 4.5 ml HCl to each tube, mix and stand at room temperature for another hour. Digest in a hot block for 1 hour at 40°C, 1 hour at 60°C, 1 hour at 80°C and 4 hrs at 100°C allowing 1/2 hr. ramping times
NOTE: The final volume should be less than 2 ml; if more, continue heating at 125°C to accomplish this.
Dilute with DDW to 25 ml and mix thoroughly.
Settle out suspended particles overnight and decant clear supernatant into disposable plastic tubes for analysis by AES-ICP. Samples requiring 5 or more metals are analyzed by AES-ICP (see below).
INTERFERENCES: Several, which are compensated for by the computer program.
REPORTING RESULTS: Two significant figures. µg/g Sr
INSTRUMENTATION: For the determination of 5 or more elements:
Inductively coupled Plasma Emission Spectrometer- Jobin-Yvon 1 m.JY48P with Autosampler & DEC computer.PET interface to LIS.(See "Remarks").
Calibration Range: 0 - 10 mg/l.
Resolution: Four significant figures (0.0001 mg/l).
Sensitivity: Not applicable (NA).
Instrument Detection Limit:.001 mg/l.
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.05 to 100 µg/g.
Accuracy- Not determined (ND).
Precision of Controls-

	A	B
mean	56 µg/g	
std. dev.	3.7 µg/g	
R.S.D.	6.6 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	0.84	1.9	1.9
mean	13	33	76

W 0.5 µg/g T 2.5 µg/g

CONTROL LIMITS: µg/g

Control		W.L. (x ±2σ)	R.L. (x ±3σ)
QCRS85-1	L.L.	49	45
	U.L.	63	67

REMARKS:- Determination of boron MUST be on Atom Scan 2400 only.
- Extraction % (±) based on average recovery of 4 samples from Soil Research Institute - SO1, SO2, SO3 & SO4.
- Samples requiring only Mg, Al and/or Ca are analyzed by Atom Scan.

SUMMARY REPORT OF QUALITY CONTROL DATA

STRONTIUM IN SEDIMENT

Operating Range = 0.050 to 100.0 ug/g

IN - RUN DUPLICATES

Range	<0.050	0.050 to20.00	20.00 to50.00	50.00 to100.0	>100.0
no.	1	9	13	11	10
s.w.		0.8372	1.8808	1.8710	
mean		13.0406	32.7930	76.3727	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
QCRS85-1	121	55.861	3.7047	6.63

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
rb	35	0.055	0.0495

DATE 87/04/23

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: TITANIUM TEST CODE: TIUT SAMPLE TYPE: SEDIMENT
UNIT: Vegetation/Soil/Sediment SUPERVISOR: L. Pastorek

METHOD CODE:535AA0 HMARSOIL - ICP/AES
REVISION NO: Original DATE: 1980
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- 10 to 20 g of wet sample
Container- 500 ml PET jar
Preservative- None
Other- Samples are air-dried and ground to less than 45 mesh.

SAMPLE PREPARATION:Partial Extn.-Yes Total Extn.- % Extracted-
Procedure- Weigh 0.500g sample into a calibrated digestion tube and add 1.5 ml HNO3 . Mix and stand at room temperature for 2 hrs. Add 4.5 ml HCl to each tube, mix and stand at room temperature for another hour. Digest in a hot block for 1 hour at 40°C, 1 hour at 60°C, 1 hour at 80°C and 4 hrs at 100°C allowing 1/2 hr. ramping times
NOTE: The final volume should be less than 2 ml; if more, continue heating at 125°C to accomplish this.
Dilute with DDW to 25 ml and mix thoroughly.
Settle out suspended particles overnight and decant clear supernatant into disposable plastic tubes for analysis by AES-ICP. Samples requiring 5 or more metals are analyzed by AES-ICP (see below).
INTERFERENCES: Several, which are compensated for by the computer program.
REPORTING RESULTS: Two significant figures. µg/g Ti
INSTRUMENTATION: For the determination of 5 or more elements:
Inductively coupled Plasma Emission Spectrometer- Jobin-Yvon 1 m.JY48P with Autosampler & DEC computer.PET interface to LIS.(See "Remarks").
Calibration Range: 0 -10 mg/l.(JA)
Resolution: Four significant figures (0.0001 mg/l).
Sensitivity: Not applicable (NA).
Instrument Detection Limit:.004 mg/l.
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.2 to 1000 µg/g
Accuracy- Not determined (ND).
Precision of Controls-

	A	B
mean	580 µg/g	
std. dev.	170 µg/g	
R.S.D.	29 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	36	69	120
mean	140	320	750

W 10 µg/g T 50 µg/g

CONTROL LIMITS: µg/g

		W.L. (x ±2σ)	R.L. (x ±3σ)
Control	L.L.	240	70
QCRS85-1	U.L.	920	1100

REMARKS:- Determination of boron MUST be on Atom Scan 2400 only.
- Extraction % (±) based on average recovery of 4 samples from Soil Research Institute - S01, S02, S03 & S04.
- Samples requiring only Mg, Al and/or Ca are analyzed by Atom Scan.

SUMMARY REPORT OF QUALITY CONTROL DATA

TITANIUM IN SEDIMENT

Operating Range = 0.200 to 1000.0 ug/g

IN - RUN DUPLICATES

Range	<0.200	0.200 to200.00	200.00to500.00	500.00to1000.0	>1000.0
no.	4	10	16	11	3
s.w.		35.6806	68.7456	116.7282	
mean		134.5044	325.0567	750.0438	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
QCRS85-1	122	577.237	166.9015	28.91

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
rb	55	1.155	2.6850

DATE 87/04/23

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: VANADIUM TEST CODE: VVUT SAMPLE TYPE: SEDIMENT
UNIT: Vegetation/Soil/Sediment SUPERVISOR: L. Pastorek

METHOD CODE:535AA0 HMARSOIL - ICP/AES
REVISION NO: Original DATE: 1980
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- 10 to 20 g of wet sample
Container- 500 ml PET jar
Preservative- None
Other- Samples are air-dried and ground to less than 45 mesh.

SAMPLE PREPARATION:Partial Extn.-Yes Total Extn.- % Extracted-65±10
Procedure- Weigh 0.500g sample into a calibrated digestion tube and add 1.5 ml HNO3 . Mix and stand at room temperature for 2 hrs. Add 4.5 ml HCl to each tube, mix and stand at room temperature for another hour. Digest in a hot block for 1 hour at 40°C, 1 hour at 60°C, 1 hour at 80°C and 4 hrs at 100°C allowing 1/2 hr. ramping times
NOTE: The final volume should be less than 2 ml; if more, continue heating at 125°C to accomplish this.
Dilute with DDW to 25 ml and mix thoroughly.
Settle out suspended particles overnight and decant clear supernatant into disposable plastic tubes for analysis by AES-ICP. Samples requiring 5 or more metals are analyzed by AES-ICP (see below).
INTERFERENCES: Several, which are compensated for by the computer program.
REPORTING RESULTS: Two significant figures. µg/g V
INSTRUMENTATION: For the determination of 5 or more elements:
Inductively coupled Plasma Emission Spectrometer- Jobin-Yvon 1 m.JY48P with Autosampler & DEC computer.PET interface to LIS.(See "Remarks").
Calibration Range: 0 - 10 mg/l.
Resolution: Four significant figures (0.0001 mg/l).
Sensitivity: Not applicable (NA).
Instrument Detection Limit:.01 mg/l.
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.5 to 100 µg/g.
Accuracy- Not determined (ND).
Precision of Controls-

	A	B
mean	34 µg/g	
std. dev.	2.1 µg/g	
R.S.D.	6.3 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	1.5	1.5	2.0
mean	13	34	68

W .5 µg/g T 2.5 µg/g

CONTROL LIMITS: µg/g		W.L. (x ±2σ)	R.L. (x ±3σ)
Control	L.L.	30	28
QCRS85-1	U.L.	38	40

REMARKS:- Determination of boron MUST be on Atom Scan 2400 only.
- Extraction % (±) based on average recovery of 4 samples from Soil Research Institute - SO1, SO2, SO3 & SO4.
- Samples requiring only Mg, Al and/or Ca are analyzed by Atom Scan.

SUMMARY REPORT OF QUALITY CONTROL DATA

VANADIUM IN SEDIMENT

Operating Range = 0.500 to 100.0 ug/g

IN - RUN DUPLICATES

Range	<0.500	0.500 to20.00	20.00 to50.00	50.00 to100.0	>100.0
no.	1	11	27	5	0
s.w.		1.4961	1.4954	1.9831	
mean		13.1952	34.0391	67.6908	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
QCRS85-1	122	33.730	2.1247	6.30

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
rb	47	0.275	0.2500

DATE 87/04/23

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: ZINC TEST CODE: ZNUT SAMPLE TYPE: SEDIMENT
UNIT: Vegetation/Soil/Sediment SUPERVISOR: L. Pastorek

METHOD CODE:535AA0 HMARSOIL - ICP/AES
REVISION NO: Original DATE: 1980
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- 10 to 20 g of wet sample
Container- 500 ml PET jar
Preservative- None
Other- Samples are air-dried and ground to less than 45 mesh.

SAMPLE PREPARATION:Partial Extn.-Yes Total Extn.- % Extracted-85±5
Procedure- Weigh 0.500g sample into a calibrated digestion tube and add 1.5 ml HNO3 . Mix and stand at room temperature for 2 hrs. Add 4.5 ml HCl to each tube, mix and stand at room temperature for another hour. Digest in a hot block for 1 hour at 40°C, 1 hour at 60°C, 1 hour at 80°C and 4 hrs at 100°C allowing 1/2 hr. ramping times
NOTE: The final volume should be less than 2 ml; if more, continue heating at 125°C to accomplish this.
Dilute with DDW to 25 ml and mix thoroughly.
Settle out suspended particles overnight and decant clear supernatant into disposable plastic tubes for analysis by AES-ICP. Samples requiring 5 or more metals are analyzed by AES-ICP (see below).
INTERFERENCES: Several, which are compensated for by the computer program.
REPORTING RESULTS: Two significant figures. µg/g Zn
INSTRUMENTATION: For the determination of 5 or more elements:
Inductively coupled Plasma Emission Spectrometer- Jobin-Yvon 1 m.JY48P with Autosampler & DEC computer.PET interface to LIS.(See "Remarks").
Calibration Range: 0 - 10 mg/l.
Resolution: Four significant figures (0.0001 mg/l).
Sensitivity: Not applicable (NA).
Instrument Detection Limit: .02 mg/l.
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 1 to 500 µg/g.
Accuracy- Not determined (ND).
Precision of Controls-

	A	B
mean	3000 µg/g	
std. dev.	290 µg/g	
R.S.D.	9.7 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	5.29	6.1	18
mean	55	150	390

W 2 µg/g T 10 µg/g

CONTROL LIMITS: µg/g

Control		W.L. (x ±2σ)	R.L. (x ±3σ)
QCRS85-1	L.L.	2400	2100
	U.L.	3600	3900

REMARKS:- Determination of boron MUST be on Atom Scan 2400 only.
- Extraction % (±) based on average recovery of 4 samples from Soil Research Institute - SO1, SO2, SO3 & SO4.
- Samples requiring only Mg, Al and/or Ca are analyzed by Atom Scan.

SUMMARY REPORT OF QUALITY CONTROL DATA

ZINC IN SEDIMENT

Operating Range = 1.000 to 500.0 ug/g

IN - RUN DUPLICATES

Range	<1.000	1.000 to100.00	100.00to250.00	250.00to500.0	>500.0
no.	2	22	9	5	6
s.w.		5.1924	6.1020	17.7496	
mean		54.8895	150.6528	392.1186	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
QCRS85-1	122	3016.723	293.6362	9.73

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
rb	92	7.320	19.200

DATE 87/04/23

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME:CARBONATE TEST CODE:CCO3UT SAMPLE TYPE:SEDIMENT
UNIT:Veg/Soil/Sed SUPERVISOR: L. Pastorek

METHOD CODE:001BP5 in CARBONAT
REVISION NO: DATE:
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required-10 to 20 g of wet sample
Container- 500 ml PET jar
Preservative- none
Other- Sample is air-dried and ground to less than 45 mesh

SAMPLE PREPARATION:Partial Extn.- Total Extn.- % Extracted-
Procedure- Place approx. 0.1g of sample (weighed on 4 dec. balance) into a impinger tube. Add enough DDW to wet sample and place tube in sampling train. Turn on pump to pass air through the NaOH scrubber, the sample impinger, the KI solution and then into the coulometric cell. Add 2ml of 2N HCl to the sample and turn on the heater. The CO2 evolved is carried along the sampling train by the NaOH scrubbed air to the coulometric cell where it is automatically titrated. After 10 min. the coulometer has completed it titration of the CO2 evolved and the display on the coulometer gives the amount of µg of carbonate carbon measured by the cell.

INTERFERENCES: oxidation of organic matter

REPORTING RESULTS: % dry wt to two significant figures.
INSTRUMENTATION:Coulometrics 5010 CO2 coulometer, Coulometrics 5030 carbonate carbon impinger train.

Calibration Range: .01 to 3000 µg of carbonate carbon
Resolution:0.01 µg of C
Sensitivity: ND
Instrument Detection Limit: 5 µg of C

PERFORMANCE CHARACTERISTICS:
Routine Operating Range-.01 to 5% carbon
Accuracy- 100% ±3% for 0.02g of BaCO3
Precision of Controls-

	A	B
mean	.766 %	6.07 %
std. dev.	.024 %	.073 %
R.S.D.	3.2 %	1.2 %

Precision of Duplicates-

	low range	mid range	high range
s.d.	.0322	.135	.0608
mean	.149	1.96	3.53

W .02 % T .10 %

CONTROL LIMITS: %	A W.L. (x ±2σ)	B W.L. (x ±2σ)	A R.L. (x ±3σ)	B R.L. (x ±3σ)
L.L.	.72	6.0	.70	5.9
U.L.	.82	6.2	.84	6.3

REMARKS: Sample weight is selected so that 1000 to 3000 µg of C is evolved. Calculation -% carbonate C =(µg of C - blk)/(wt in g X 10000)
Control A = QCRS85-1
Control B = BaCO3

SUMMARY REPORT OF QUALITY CONTROL DATA

CARBONATE IN SEDIMENT

Operating Range = 0.010 to 5.0 %carbon

IN - RUN DUPLICATES

Range	<0.010	0.010 to1.00	1.00 to2.50	2.50 to5.0	>5.0
no.	0	7	11	7	1
s.w.		0.0322	0.1346	0.0608	
mean		0.1493	1.9595	3.5313	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
rs85-1	36	0.766	0.0241	3.15
BACO3	43	6.072	0.0732	1.21

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLK	76	8.634	1.8361

DATE 87/04/28

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME:TOTAL CARBON TEST CODE:CCUT SAMPLE TYPE:SEDIMENT
UNIT:Veg/Soil/Sed SUPERVISOR: L. Pastorek

METHOD CODE:001BF7 in PHYSOLID
REVISION NO: DATE:
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- 10 to 20 g of wet sample
Container- 500 ml PET jar
Preservative- none
Other- Sample is air-dried and ground to less than 45 mesh

SAMPLE PREPARATION:Partial Extn.- Total Extn.- % Extracted-
Procedure- Place between .1 and .5g of ground sample in a tared boat and enter weight of sample in weight stack. Place sample in furnace and combust at 1370 °C in a oxygen atmosphere. The CO2 evolved is collected and passed through several filters to absorb impurities. The sample travels along the train to an infrared detector where the CO2 is measured and the % carbon calculated by the instruments microprocessor.

INTERFERENCES:moisture and dust

REPORTING RESULTS: % dry
INSTRUMENTATION: CR12 LECO Carbon Determinator

Calibration Range: .01 to 100%
Resolution:.01 %
Sensitivity: ND
Instrument Detection Limit: 0.01 %

PERFORMANCE CHARACTERISTICS:
Routine Operating Range-.01 to 10% carbon
Accuracy- ND
Precision of Controls-

	A	B
mean	3.4 %	12.2 %
std. dev.	.089 %	.42 %
R.S.D.	2.7 %	3.4 %

Precision of Duplicates-

	low range	mid range	high range
s.d.	.041	.12	.10
mean	1.3	3.2	7.3

W .02 % T .10 %

CONTROL LIMITS: %	A W.L. (x ±2σ)	B W.L. (x ±2σ)	A R.L. (x ±3σ)	B R.L. (x ±3σ)
L.L.	3.2	11	3.1	11
U.L.	3.6	13	3.7	13

REMARKS: The product of sample weight (grams) and % carbon should not exceed 10. -i.e. 10%C at 1g, 20%C at .5g, 40% at .25g
Control A = QCRS85-1
Control B = CaCO3

SUMMARY REPORT OF QUALITY CONTROL DATA

TOTAL CARBON IN SEDIMENT

Operating Range = 0.010 to 10.0 %carbon

IN - RUN DUPLICATES

Range	<0.010	0.010 to2.00	2.00 to5.00	5.00 to10.0	>10.0
no.	0	10	18	16	25
s.w.		0.0412	0.1190	0.1003	
mean		1.2979	3.1843	7.2812	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
rs85-1	29	3.349	0.0891	2.66
CACO3	49	12.213	0.4180	3.42

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
blk	0	0	0

DATE 87/01/05

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: CHLORIDE TEST CODE: CLEW SAMPLE TYPE:SEDIMENT
UNIT: Vegetation/Soil/Sediment SUPERVISOR: L. Pastorek

METHOD CODE:305BI5 CLSOILS
REVISION NO: 1 DATE: Sept.,1987
NATURE OF LAST REVISION: From alkaline fusion to water extraction.

SAMPLE HANDLING:
Quantity Required- 10 to 20 g of wet sample
Container- 500 ml PET jar
Preservative- None
Other- Samples are air-dried and ground to less than 45 mesh

SAMPLE PREPARATION:Partial Extn.-Yes Total Extn.- % Extracted-
Procedure- Place 3.0g of air-dried sample in a 50ml centrifuge tube. Add 30 ml distilled water and shake for 1 hour on a reciprocating shaker. Centrifuge, filter through a membrane filter and analyze for water-extractable chloride by ion chromatography.

INTERFERENCES:

REPORTING RESULTS:Two significant figures (µg/g Cl)
INSTRUMENTATION: Ion chromatograph.

Calibration Range: 0 to 100 mg/l.
Resolution: 0.1 mg/l).
Sensitivity:
Instrument Detection Limit: 1 mg/l.

PERFORMANCE CHARACTERISTICS:
Routine Operating Range-.01 to 100 µg/g.
Accuracy- Not determined (ND).
Precision of Controls-

	A	B
mean	46 µg/g	14
std. dev.	2.8 µg/g	3.4
R.S.D.	6 %	25 %

Precision of Duplicates-

	low range	mid range	high range
s.d.	1.8	NA	NA
mean	9.7	NA	NA

W 1 µg/g T 5 µg/g

CONTROL LIMITS: µg/g

Control		W.L. (x ±2σ)	R.L. (x ±3σ)
	L.L.	8.6	5.4
	U.L.	21	25

REMARKS:- Three controls - A, B, & C.
- > ± 3 sd (standard deviations) on digested control samples before rejection of run.

SUMMARY REPORT OF QUALITY CONTROL DATA

EXTRACTABLE CHLORIDE IN SEDIMENT

Operating Range = 0.010 to 100.0 ug/g

IN - RUN DUPLICATES

Range	<0.010	0.010 to20.00	20.00 to50.00	50.00 to100.0	>100.0
no.	0	2	0	0	4
s.w.		0.0200	0.0000	0.0000	
mean		1.1700	0.0000	0.0000	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
soil	6	46.370	2.7670	5.97
clay	6	13.680	3.4290	25.07
clay	13	14.900	3.1780	21.33

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
blk	0	0.000	0.0000

DATE 87/04/23

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Total cyanide TEST CODE: CCNAUR SAMPLE TYPE:SEDIMENT
UNIT:Project-QC SUPERVISOR: J. Hipfner

METHOD CODE:302AC2
REVISION NO: DATE:
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- 10 to 20 g of wet sample
Container- 500 ml PET jar
Preservative- None
Other- Sample is air-dried and ground to less than 45 mesh

SAMPLE PREPARATION:Partial Extn.- Total Extn.- % Extracted-100
Procedure- 5g of wet sample is extracted into 50 ml of water and the extract analyzed by the automated high temperature distillation with 25%H3PO4, 5%H3PO2 followed by a colourimetric analysis with the chloramine T -isonicotinic acid -barbituric acid method.
If the total cyanide is > .1 µg/g, then 5g of sample, 250 ml of DDW water and 30 ml of 15%(w/v) tartaric acid are distilled by a manual distillation procedure. The distillate is collected in 50 ml of 1N NAOH, and analyzed using the automated system referred to above.

INTERFERENCES: SCN interference is removed by distillation. Distillable organics may interfere; also sulfide at high levels.
REPORTING RESULTS:Mg/l CN: 3 decimal places up to 3 significant figs
INSTRUMENTATION: Technicon AAII continuous flow analyzer including pump, colourimeter, appropriate autosampler and recorder. High temperature distillation apparatus (Technicon). Manual dist. app
Calibration Range: 0 to 0.4 mg/l as CN
Resolution: 0.001 mg/l
Sensitivity:
Instrument Detection Limit: 0.001 mg/l
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.01 to 4.00 ug/g
Accuracy- 100%
Precision of Controls-

	A	B
mean	1.1 ug/g	.59 ug/g
std. dev.	0.027ug/g	0.026ug/g
R.S.D.	2.4 %	4.4 %

Precision of Duplicates-low range mid range high range
s.d.
mean
W 0.01 µg/g T 0.05 µg/g

CONTROL LIMITS:

REMARKS: Pure CN standards are recovered 100% during manual distillation. Complex cyanides can normally be expected to be recovered at 100%.

SUMMARY REPORT OF QUALITY CONTROL DATA

TOTAL CYANIDE IN SEDIMENT

Operating Range = 0.01 to 4 ug/g

IN - RUN DUPLICATES

Range	<0.01	0.01 to0.8	0.8 to2	2 to4	>4
no.	0	2	0	0	0
s.w.		0	0	0	
mean		0.01	0	0	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
qc-a	146	1.49	0.049	3.29
qc-b	146	0.18	0.022	12.22

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLK	146	0.01	0

DATE 88/06/01

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Free cyanide TEST CODE: CCNFUR SAMPLE TYPE:SEDIMENT
UNIT:Project-QC SUPERVISOR: J. Hipfner

METHOD CODE:302AC2
REVISION NO: DATE:
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- 10 to 20 g of wet sample
Container- 500 ml PET jar
Preservative- None
Other- Sample is air-dried and ground to less 45 mesh.

SAMPLE PREPARATION:Partial Extn.- Total Extn.- % Extracted-100
Procedure- 5g of the wet sample is extracted into 50 ml of distilled water by shaking for .5 hrs. and the extract is made alkaline by addition of .1 ml of 10N NaOH. The extract is then centrifuged and the clear supernatent is passed through an automated low temperature dist. (106°C) in a distillation acid consisting of 10% acetic acid and .5% zinc acetate. The distillate recovered is analyzed by the chloramine T isonicotinic acid-barbituric acid colourimetric method

INTERFERENCES: None

REPORTING RESULTS:Mg/l CN to 2 decimal places up to 3 significant figs
INSTRUMENTATION: Technicon automated continuous flow analyzer including pump, colourimetric distillation apparatus and sampler; suitable recorder.
Calibration Range: 0 to 0.4 mg/l as CN
Resolution: 0.001
Sensitivity:
Instrument Detection Limit: 0.001 mg/l
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.0100 to 4.00 ug/g
Accuracy- 100%
Precision of Controls-

	A	B
mean	1.10 ug/g	0.60 ug/g
std. dev.	.036 ug/g	.031 ug/g
R.S.D.	3.3 %	5.2 %

Precision of Duplicates-low range mid range high range
s.d.
mean
W 0.01 µg/g T 0.05 µg/g

CONTROL LIMITS:

REMARKS:* The test defines the results reported in this case. The terminology "Weak Acid Dissociable" is commonly used and represents weakly associated cyanide compounds such as KCN, NaCN, NiCN4, HCN,etc.

SUMMARY REPORT OF QUALITY CONTROL DATA

FREE CYANIDE IN SEDIMENT

Operating Range = 0.01 to 4 ug/g

IN - RUN DUPLICATES

Range	<0.01	0.01 to0.8	0.8 to2	2 to4	>4
no.	0	2	0	0	0
s.w.		0	0	0	
mean		0.01	0	0	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
qc-a	135	1.51	0.062	4.11
qc-b	135	0.18	0.022	12.22

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLK	135	0.01	0

DATE 88/06/01

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: FLUORIDE TEST CODE: FFUT SAMPLE TYPE:SEDIMENT
UNIT: Vegetation/Soil/Sediment SUPERVISOR: L. Pastorek

METHOD CODE:550APO
REVISION NO: Original DATE: 1983, Sept.,1987
NATURE OF LAST REVISION: Update sheets.

SAMPLE HANDLING:
Quantity Required-10 to 20 g of wet sample
Container- 500 ml PET jar
Preservative- None
Other- Samples are air-dried and ground to less than 45 mesh

SAMPLE PREPARATION:Partial Extn.-Yes Total Extn.- % Extracted-
Procedure- Extract 0.500g sample for 4 hrs. with 25 ml of 0.1N HClO4 in a water bath at 80C. Place samples on on shaker for 1 hour. Remove and dilute to 50 ml. with TISAB III buffer solution. Mix well, and allow to stand overnight to complete extration and complexation process prior to analysis by fluoride ion selective electrode.

INTERFERENCES: High concentrations of Al (> 500 ug/g), Ca, Mg, and Fe
OH- and H+ ions, pH must be adjusted to 5.5
REPORTING RESULTS: Two significant figures (ug/g F).
INSTRUMENTATION: Orion Research Model 901; microprocessor Ionalyzer with Orion 9609 combination fluoride electrode.

Calibration Range: 0 - 5 ug/l.
Resolution: 0.01 ug/l).
Sensitivity:
Instrument Detection Limit: 0.01 ug/l.

PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.2 to 500 µg/g.
Accuracy- Not determined (ND).
Precision of Controls-

	A	B
mean	154 µg/g	42
std. dev.	7 µg/g	3
R.S.D.	5 %	7%

Precision of Duplicates-

	low range	mid range	high range
s.d.	0.72	NA	NA
mean	27	NA	NA

W 0.5 µg/g T 2.5 µg/g

CONTROLS	A	B	A	B
CONTROL LIMITS: µg/g	W.L. (x ±2σ)		R.L. (x ±3σ)	
Control L.L.	16	36	14	33
U.L.	24	48	26	51

REMARKS:-Control A -New Soil
Control B -ref#3

SUMMARY REPORT OF QUALITY CONTROL DATA

FLUORIDE IN SEDIMENT

Operating Range = .2 to 500 ug/g

IN - RUN DUPLICATES

Range	<.2	.2 to100	100 to250	250 to 500	> 500
no.					
s.w.					
mean					

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
veggc	6	154.500	7.2590	4.70
soilqc	6	19.550	2.0790	10.63
REF#3	4	41.500	2.9500	7.11

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
blk	0	0.000	0.0000

DATE 87/01/06

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME:LOSS ON IGN. TEST CODE:RSTLOI SAMPLE TYPE:SEDIMENT
UNIT:Veg/Soil/Sed SUPERVISOR: L. Pastorek

METHOD CODE:571CI4 in PHYSOLID
REVISION NO: DATE:
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- 10 to 20 g of wet sample
Container- 500 ml PET jar
Preservative-
Other- Samples are air-dried and ground to less than 45 mesh

SAMPLE PREPARATION:Partial Extn.- Total Extn.- % Extracted-
Procedure- Weigh approximately 1 g of sample into a tared crucible. Dry sample in an oven at 105°C for 16 hrs and reweigh. Place the crucible and the sample in a muffle furnace and ash the sample for 4 hrs at 475°C. Remove from furnace, cool, and weigh.

INTERFERENCES:

REPORTING RESULTS: mg/g to two significant figures.
INSTRUMENTATION: Balance and muffle furnace

Calibration Range:NA
Resolution:.01 g
Sensitivity:
Instrument Detection Limit:.01g

PERFORMANCE CHARACTERISTICS:
Routine Operating Range-.1 to 100 mg/g
Accuracy-
Precision of Controls-

	A	B
mean	42.7 mg/g	
std. dev.	3.05 mg/g	
R.S.D.	7.1 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	.85	.64	9.47
mean	12.9	36.4	75.5

W .5 mg/g T 2.5 mg/g

CONTROL LIMITS: mg/g

	W.L.(x ±2σ)	R.L.(x ±3σ)
L.L.	37	34
U.L.	49	52

REMARKS:%LOI={(wt after drying-wt after ashing)/wt after drying}X100%
N.B.- Subtract tare wt of crucible from above weights

SUMMARY REPORT OF QUALITY CONTROL DATA

LOSS ON IGNITION IN SEDIMENT

Operating Range = 0.100 to 100.0 mg/g

IN - RUN DUPLICATES

Range	<0.100	0.100 to20.00	20.00 to50.00	50.00 to100.0	>100.0
no.	0	6	2	3	4
s.w.		0.8505	0.6403	9.4685	
mean		12.8742	36.4000	75.4667	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
rs85-1	24	42.704	3.0482	7.14

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
blk	0	0	0

DATE 87/04/23

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME:MOISTURE TEST CODE:MOIST SAMPLE TYPE:SEDIMENT
UNIT:Veg/Soil/Sed SUPERVISOR: L. Pastorek

METHOD CODE:002BI4 in PHYSOLID
REVISION NO: DATE:
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- 10 to 20 g of wet sample
Container- 500ml PET jar
Preservative-none
Other-

SAMPLE PREPARATION:Partial Extn.- Total Extn.- % Extracted-
Procedure- Dry evaporating dish at 100 °C for 2 hrs., cool in desiccator, and weigh to nearest .01g. Transfer 5 to 10 g of wet sample into the dish and weigh immediately. Evaporate sample for 16 hrs. at 105°C. Reweigh sample after allowing time for sample to cool in a desiccator.

INTERFERENCES:volatile materials which evaporate at 105°C

REPORTING RESULTS: per cent to two significant figures.
INSTRUMENTATION:

Calibration Range:NA
Resolution:.01 g
Sensitivity:NA
Instrument Detection Limit:.01g

PERFORMANCE CHARACTERISTICS:
Routine Operating Range-.01 to 100% moisture
Accuracy-
Precision of Controls-

	A	B
mean	%	%
std. dev.	%	%
R.S.D.	%	%

Precision of Duplicates-

	low range	mid range	high range
s.d.	.68	3.01	.57
mean	17.1	38.6	72.5

W T

CONTROL LIMITS:

REMARKS: % moisture = {(wet wt - dry wt) / wet wt} X 100 %
N.B. Subtract out tare weight of dish.

SUMMARY REPORT OF QUALITY CONTROL DATA

MOISTURE IN SEDIMENT

Operating Range = 0.010 to 100.0 %moisture

IN - RUN DUPLICATES

Range	<0.010	0.010 to20.00	20.00 to50.00	50.00 to100.0	>100.0
no.	0	4	7	7	0
s.w.		0.6800	3.0100	0.5700	
mean		17.0500	38.5500	72.5400	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.

DATE 87/05/14

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME:NITROGEN-TOTAL TEST CODE: NNTKUR SAMPLE TYPE:SEDIMENT
UNIT: Vegetation/Soil/Sediment SUPERVISOR: L. Pastorek

METHOD CODE: TYPE:Acid digestion/automated colourimetry
REVISION NO: #1 DATE: Sept. 1987.
NATURE OF LAST REVISION: Update of QC data.

SAMPLE HANDLING:
Quantity Required- 10 to 20 g of wet sample
Container- 500 ml PET jar
Preservative- None
Other- Samples are air-dried and ground to less than 45 mesh

SAMPLE PREPARATION:Partial Extn.- Total Extn.-Yes % Extracted-
Procedure- Digest sample aliquot with 7 ml of H_2SO_4 for about 2 hrs until intense fuming occurs. Add 2 g potassium persulphate and take to fuming until the digestate is clear. Cool and make up digestate to 100 ml. Titrate a 25 ml aliquot with 6.25N NaOH to pH 12 and filter out precipitated metals.
Add 8 ml of 4% EDTA solution, adjust the pH with 10% H_2SO_4 to the methyl red end point (pH 4) and adjust the volume to 100 ml. Analyze for nitrogen using the automated phenate method.

INTERFERENCES:

REPORTING RESULTS:Two significant figures mg/g N
INSTRUMENTATION: Automated colourimetric system (Technicon or equivalent) with 630 nm filters.

Calibration Range: 0 to 10 mg/l NH_3 as N.
Resolution: 0.01 mg/l.
Sensitivity:
Instrument Detection Limit: 0.05 mg/l.

PERFORMANCE CHARACTERISTICS:
Routine Operating Range-0.02 to 5 mg/g N dry wt. for 0.2 g sample
Accuracy- ND
Precision of Controls-

	A	B
mean	1.2 mg/g	
std. dev.	0.06 mg/g	
R.S.D.	4.9 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	.015	.038	0.14
mean	.17	1.5	3.3

W 0.05 mg/g T .25 mg/g

CONTROL LIMITS: mg/g

Control		W.L. (x ±2σ)	R.L. (x ±3σ)
	L.L.	1.1	1.0
	U.L.	1.3	1.4

REMARKS:- Control limits:

- Precision based on 0.2 g sample.
- Total phosphorus (PPUT) also on the same sample digest.
- Precision of duplicates - other values for sediments.

SUMMARY REPORT OF QUALITY CONTROL DATA

NITROGEN IN SEDIMENT

Operating Range = 0.020 to 5.0 mg/g

IN - RUN DUPLICATES

Range	<0.020	0.020 to1.00	1.00 to2.50	2.50 to5.0	>5.0
no.	0	4	4	1	2
s.w.		0.0150	0.0380	0.1410	
mean		0.1700	1.4700	3.2800	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
refa	36	1.178	0.0582	4.94

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLK	0	0.000	0.0000

DATE 87/02/02

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME:PHOSPHORUS-TOT TEST CODE: PPUT SAMPLE TYPE:SEDIMENT
UNIT: Vegetation/Soil/Sediment SUPERVISOR: L. Pastorek

METHOD CODE: TYPE:Acid digestion/automated colourimetry
REVISION NO: Original #1 DATE: 1970
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- 10 to 20 g wet sample
Container- 500 ml PET jar
Preservative- None
Other- Samples are air-dried and ground to less than 45 mesh

SAMPLE PREPARATION:Partial Extn.- Total Extn.-Yes % Extracted-
Procedure- Digest sample aliquot with 7 ml of H_2SO_4 for about 2 hrs until intense fuming occurs. Add 2 g potassium persulphate and take to fuming until the digestate is clear. Cool and make up digestate to 100 ml. Titrate a 25 ml aliquot with 6.25N NaOH to pH 12 and filter out precipitated metals.
Add 8 ml of 4% EDTA solution, adjust the pH with 10% H_2SO_4 to the methyl red end point (pH 4) and adjust the volume to 100 ml. Analyze phosphorus using the automated stannous chloride reduced phosphomolybdate procedure.

INTERFERENCES: Heavy metal interferences removed by precipitated filtration.
REPORTING RESULTS:Two significant figures. mg/g P
INSTRUMENTATION: Automated colourimetric system (Technicon or equivalent) with 660 nm filters.

Calibration Range: 0 to 2 mg/l PO_4 as P.
Resolution: 0.002 mg/l.
Sensitivity:
Instrument Detection Limit: 0.01 mg/l.

PERFORMANCE CHARACTERISTICS:
Routine Operating Range-.005 to 1 mg/g PO_4 as P (for 0.2 g).
Accuracy- NBS reference not available for these matrices.
Precision of Controls-

	A	B
mean	0.89 mg/g	
std. dev.	0.031mg/g	
R.S.D.	3.4 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	.022	.007	.043
mean	.15	.23	.65

W .01 mg/g T .05 mg/g

CONTROL LIMITS: mg/g

Control		W.L. (x ±2σ)	R.L. (x ±3σ)
	L.L.	0.83	0.80
	U.L.	0.95	0.98

REMARKS:- Control limits:
- > ± 3 sd (standard deviations) on digested control samples before rejection of run.
- Precision based on 0.2 g sample.

SUMMARY REPORT OF QUALITY CONTROL DATA

PHOSPHORUS IN SEDIMENT

Operating Range = 0.005 to 1.0 mg/g

IN - RUN DUPLICATES

Range	<0.005	0.005 to0.20	0.20 to0.50	0.50 to1.0	>1.0
no.	0	3	1	3	4
s.w.		0.0220	0.0070	0.0430	
mean		0.1500	0.2300	0.6500	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
refa	36	0.887	0.0306	3.45

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLK	0	0.000	0.0000

DATE 87/02/02

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: ALUMINUM TEST CODE: ALUT SAMPLE TYPE: SOIL
UNIT: Vegetation/Soil/Sediment SUPERVISOR: L. Pastorek

METHOD CODE:535BA0 - ICP/AES -HMPNSOIL (JY)
REVISION NO: Original DATE: 1980
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- Approximately 20 g
Container- Glass jars (4oz) with bakelite screw cap.
Preservative- None
Other- Samples are air-dried and ground to less than 45 mesh.

SAMPLE PREPARATION:Partial Extn.-Yes Total Extn.- % Extracted-34±10
Procedure- Weigh 0.500g sample into a calibrated digestion tube and add 1.5 ml HNO3 . Mix and stand at room temperature for 2 hrs. Add 4.5 ml HCl to each tube, mix and stand at room temperature for another hour. Digest in a hot block for 1 hour at 40°C, 1 hour at 60°C 1 hour at 80°C and 4 hrs at 100°C allowing 1/2 hr. ramping times
NOTE: The final volume should be less than 2 ml; if more, continue heating at 125°C to accomplish this.
Dilute with DDW to 25 ml and mix thoroughly.
Settle out suspended particles overnight and decant clear supernatant into disposable plastic tubes for analysis by AES-ICP. Samples requiring 5 or more metals are analyzed by AES-ICP (see below).
INTERFERENCES: Several, which are compensated for by the computer program.
REPORTING RESULTS: Two significant figures.
INSTRUMENTATION: For the determination of 5 or more elements:
Inductively coupled Plasma Emission Spectrometer- Jobin-Yvon 1 m.JY48P with Autosampler & DEC computer.PET interface to LIS.(See "Remarks").
Calibration Range: 0 - 50 mg/l.(JA)
Resolution: Four significant figures (0.0001 mg/l).
Sensitivity: Not applicable (NA).
Instrument Detection Limit:.0791 mg/l.
PERFORMANCE CHARACTERISTICS:
Routine Operating Range-4 to 20000 µg/g.
Accuracy- Not determined (ND).
Precision of Controls-

	A	B
mean	11000µg/g	
std. dev.	540 µg/g	
R.S.D.	5.0 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	150	460	700
mean	3100	8200	13000

W 20 µg/g T 100 µg/g

CONTROL LIMITS: µg/g

Control		W.L. (x ±2σ)	R.L. (x ±3σ)
QCRS85-1	L.L.	9900	9400
	U.L.	12000	13000

REMARKS:- Determination of boron MUST be on Atom Scan 2400 only.
- Extraction % (±) based on average recovery of 4 samples from Soil Research Institute - SO1, SO2, SO3 & SO4.
- Samples requiring only Mg, Al and/or Ca are analyzed by Atom Scan.
- Al can also be determined by Atom Scan.

SUMMARY REPORT OF QUALITY CONTROL DATA

ALUMINUM IN SOIL

Operating Range =4.0000to 20000. ug/g

IN-RUN DUPLICATES

Range	<4.0000	4.0000to4000.0	4000.0to10000.	10000.to20000.	>20000.
no.	1	4	26	35	2
s.w.		147.9960	463.7580	702.3300	
mean		3141.960	8239.630	13275.04	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
QCRS85-1	132	10830.41	545.1460	5.03

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
rb	72	0.082	0.0924

DATE 87/03/31

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: ALUMINUM TEST CODE: ALUT SAMPLE TYPE: SOIL
UNIT: Vegetation/Soil/Sediment SUPERVISOR: L. Pastorek

METHOD CODE:535BA0 - HMPNSOIL - ICP/AES (AS)
REVISION NO: Original DATE: 1980
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- Approximately 20 g
Container- Glass jars (4 oz) with bakelite screw cap.
Preservative- None
Other- Samples are air-dried and ground to less than 45 mesh.

SAMPLE PREPARATION:Partial Extn.-Yes Total Extn.- % Extracted-34±10
Procedure- Weigh 0.500g sample into a calibrated digestion tube and add 1.5 ml HNO_3 . Mix and stand at room temperature for 2 hrs. Add 4.5 ml HCl to each tube, mix and stand at room temperature for another hour. Digest in a hot block for 1 hour at 40°C, 1 hour at 60°C 1 hour at 80°C and 4 hrs at 100°C allowing 1/2 hr. ramping times
NOTE: The final volume should be less than 2 ml; if more, continue heating at 125°C to accomplish this.
Dilute with DDW to 25 ml and mix thoroughly.
Settle out suspended particles overnight and decant clear supernatant into disposable plastic tubes for analysis by AES-ICP. Samples requiring 5 or more metals are analyzed by AES-ICP (see below).
INTERFERENCES: Several, which are compensated for by the computer program.
REPORTING RESULTS: Two significant figures. (µg/g Al)
INSTRUMENTATION: For the determination of Ca, Mg, Al and/or B Inductively coupled Plasma Emission Spectrometer- Atom Scan 2400 with autosampler and computer.PET interface to LIS.(See "Remarks").
Calibration Range: 0 -50 mg/l.
Resolution: Four significant figures (0.0001 mg/l).
Sensitivity: Not applicable (NA).
Instrument Detection Limit:1.0 mg/l.
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 20 to 20000 ug/g.
Accuracy- Not determined (ND).
Precision of Controls-

	A	B
mean	13000ug/g	
std. dev.	1000 ug/g	
R.S.D.	8.0 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	ND	ND	120
mean	ND	ND	11000

W 20 µg/g T 100 µg/g

CONTROL LIMITS: µg/g

Control		W.L. (x ±2σ)	R.L. (x±3σ)
QCRS85-1	L.L.	11000	10000
	U.L.	15000	16000

REMARKS:- Determination of boron MUST be on Atom Scan 2400 only.
- Extraction % (±) based on average recovery of 4 samples from Soil Research Institute - SO1, SO2, SO3 & SO4.
- Samples requiring Al can also be analyzed by Jobin-Yvon.

SUMMARY REPORT OF QUALITY CONTROL DATA

ALUMINUM-AS IN SOIL

Operating Range =20.000to 20000. ug/g

IN-RUN DUPLICATES

Range	<20.000	20.000to4000.0	4000.0to10000.	10000.to20000.	>20000.
no.	0	0	0	1	0
s.w.		0.0000	0.0000	116.5000	
mean		0.0000	0.0000	10838.00	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
RS85-1	4	12574.35	1002.187	7.97

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BL	200	-2.975	0.0430

DATE 87/04/22

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: ALUMINUM TEST CODE: ALUT SAMPLE TYPE: SOIL
UNIT: Vegetation/Soil/Sediment SUPERVISOR: L. Pastorek

METHOD CODE:535BA0 - HMPNSOIL - AAS
REVISION NO: Original DATE: 1980
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- Approximately 20 g
Container- Glass jars (4 oz) with Bakelite screw cap.
Preservative- None
Other- Samples are air-dried and ground to less than 45 mesh.

SAMPLE PREPARATION:Partial Extn.-Yes Total Extn.- % Extracted-34±10
Procedure- Weigh 0.500g sample into a calibrated digestion tube and add 1.5 ml HNO3 . Mix and stand at room temperature for 2 hrs. Add 4.5 ml HCl to each tube, mix and stand at room temperature for another hour. Digest in a hot block for 1 hour at 40±C, 1 hour at 60°C 1 hour at 80°C and 4 hrs at 100°C allowing 1/2 hr. ramping times
NOTE: The final volume should be less than 2 ml; if more, continue heating at 125°C to accomplish this.
Add 0.5 ml 5% KCl, dilute with DDW to 25 ml and mix thoroughly. Settle out suspended particles overnight and decant clear supernatant into disposable plastic tubes for analysis. Samples requiring the determination of < 5 elements and/or Na, K or Ag are analyzed by AAS.
INTERFERENCES: Several, which may be compensated for by background correction.
REPORTING RESULTS: Two significant figures. (µg/g Al)
INSTRUMENTATION: PE 5000 atomic absorption spectrophotometer, PE Autosampler 50 and a PE Automatic Burner Controller,interfaced with Commodore Pet 4032, Tractor Printer 4022P & CBM 805C dual drive.
Calibration Range: 0 - 20 mg/l.
Resolution: 0.01 mg/l.
Sensitivity: conc. for absorbance of approx. 0.20 units (50 mg/l)
Instrument Detection Limit: 0.04 mg/l.
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 2.0 to 1000 µg/g.
Accuracy- Not determined (ND).
Precision of Controls-

	A	B
mean	12000µg/g	
std. dev.	1000 µg/g	
R.S.D.	8.0%	

Precision of Duplicates-low range mid range high range
s.d.
mean
W 20 µg/g T 100 µg/g

CONTROL LIMITS: µg/g W.L. (x ±2σ) R.L. (x ±3σ)

L.L.	10000	9000
U.L.	14000	15000

REMARKS: Control QCRS85-1
Extraction % (±) based on average recovery of 4 samples from Soil Research Institute - S01, S02, S03 & S04.

SUMMARY REPORT OF QUALITY CONTROL DATA

ALUMINUM-AA IN SOIL

Operating Range =2.0000to 1000.0 ug/g

IN-RUN DUPLICATES

Range	<2.00	2.00 to200.	200. to 500.	500.to1000.0	>1000.
no.	0	0	0	0	1
s.w.		0.0000	0.0000	0.0000	
mean		0.0000	0.0000	0.0000	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
S85-1	4	12500.00	1000.000	8.00

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.

DATE 87/04/23

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME:Antimony TEST CODE:SBUT SAMPLE TYPE:Soil
UNIT:Biomaterials SUPERVISOR: R. S. Sadana

METHOD CODE:542AF3 TYPE: Semi-aut. hydr. gen - flameless AAS
REVISION NO: Original DATE: January, 1983
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- Approximately 2 g
Container- Glass jar with bakelite screw cap
Preservative- None
Other-

SAMPLE PREPARATION:Partial Extn.- Total Extn.-Yes % Extracted->90
Procedure- Weigh 60 mg (45 mesh) sample into a 18 x 150 mm pyrex graduated test tube. Add 3 ml of acid mixture (6 nitric: 3 sulphuric: 1 perchloric). Process in batches of 80 samples including blanks, calibration standards and controls.
Digest in an aluminum hot block at a medium setting on the hot plate for 14 hrs until dense white fumes appear.
Cool, add 0.5 ml of distilled water, then add 2 ml conc. HCl.
Dilute to 15 ml with distilled water and mix.
Feed the prepared solutions to the automated system for the determination of antimony by the hydride-FAAS technique.
INTERFERENCES: Excessive concentrations of Cu, Fe and Ni may interfere.
REPORTING RESULTS:2 dec. <10, 1 dec. <100, 0 dec. if >100 µg/g
INSTRUMENTATION: Atomic absorption spectrophotometer (Varian Techtron 1200 & AA-5, with chart recorder, peristaltic pump and an autosampler.
Open ended heated quartz "T" cell (0.6x10cm); gas-liquid separator
Calibration Range:0 - 40 ng/ml (linear<20 ng/ml)
Resolution: 0.01 absorbance (unexpanded scale)
Sensitivity:0.02 µg/ml reads 0.15 abs.
Instrument Detection Limit: 0.001 µg/ml
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.3 to 10 µg/g
Accuracy- 99% (NBS orchard leaves)

Precision of Controls-

	A	B
mean	.55	18.5
std. dev.	.49	3.5
R.S.D.	89	19

Precision of Duplicates-

	low range	mid range	high range
s.d.	.19	.78	.65
mean	.80	3.06	6.59

W 0.2 µg/g T 1.0 µg/g

CONTROL LIMITS:

REMARKS:

- Detection Limit - 3x std. dev. of low range within-run duplicates.
- Accuracy - Ratio of mean to cert. value in ref. mat. X 100

SUMMARY REPORT OF QUALITY CONTROL DATA

ANTIMONY IN SOIL

Operating Range =0.3000to 10.0 ug/g

IN-RUN DUPLICATES

Range	<0.3000	0.3000to2.00	2.00 to5.00	5.00 to10.0	>10.0
no.	6	14	5	6	6
s.w.		0.2003	0.7755	0.6487	
mean		0.8720	3.0580	6.5920	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
orch leave	6	2.855	0.4465	15.64
veg contro	14	0.484	0.1798	37.15
soil cont1	34	0.554	0.4929	88.97
soil cont2	26	18.446	3.4589	18.75

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLK	0	0.000	0.0000

DATE 87/03/17

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME:Arsenic TEST CODE:ASUT SAMPLE TYPE:Soil
UNIT:Biomaterials SUPERVISOR: R. S. Sadana

METHOD CODE:542AF3 TYPE: Semi-aut. hydr. gen - flameless AAS
REVISION NO: Original DATE: January, 1983
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- Approximately 2 g
Container- Glass jar with bakelite screw cap
Preservative- None
Other-

SAMPLE PREPARATION:Partial Extn.- Total Extn.-Yes % Extracted->90
Procedure- Weigh 60 mg (45 mesh) sample into a 18 x 150 mm pyrex graduated test tube. Add 3 ml of acid mixture (6 nitric: 3 sulphuric: 1 perchloric). Process in batches of 80 samples including blanks, calibration standards and controls.
Digest in an aluminum hot block at a medium setting on the hot plate for 14 hrs until dense white fumes appear.
Cool, add 0.5 ml of distilled water, then add 2 ml conc. HCl.
Dilute to 15 ml with distilled water and mix.
Feed the prepared solutions to the automated system for the determination of arsenic by the hydride-FAAS technique.
INTERFERENCES: Excessive concentrations of Cu, Fe and Ni may interfere.
REPORTING RESULTS:2 dec. <10, 1 dec. <100, 0 dec. if >100 µg/g
INSTRUMENTATION: Atomic absorption spectrophotometer (Varian Techtron 1200 & AA-5, with chart recorder, peristaltic pump and an autosampler.
Open ended heated quartz "T" cell (0.6x10cm); gas-liquid separator
Calibration Range:0 - 40 ng/ml (linear <20ng/ml)
Resolution: 0.01 absorbance (unexpanded scale)
Sensitivity:0.02 µg/ml reads 0.15 abs.
Instrument Detection Limit: 0.001 µg/ml
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.3 to 10 µg/g
Accuracy- 99% (NBS orchard leaves)
Precision of Controls-

	A	B
mean	5.63	12.2
std. dev.	.81	1.58
R.S.D.	14	13

Precision of Duplicates-

	low range	mid range	high range
s.d.	.12	.34	.91
mean	.92	3.42	7.32

W 0.2 µg/g T 1.0 µg/g

CONTROL LIMITS:

REMARKS:

- Detection Limit - 3x std. dev. of low range within-run duplicates.
- Accuracy - Ratio of mean to cert. value in ref. mat. X 100.

SUMMARY REPORT OF QUALITY CONTROL DATA

ARSENIC IN SOIL

Operating Range =0.3000to 10.0 ug/g

IN-RUN DUPLICATES

Range	<0.3000	0.3000to2.00	2.00 to5.00	5.00 to10.0	>10.0
no.	20	34	21	22	30
s.w.		0.1188	0.3396	0.9135	
mean		0.9910	3.4160	7.3150	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
orch leave	45	9.297	1.0512	11.31
veg contro	68	9.184	1.4021	15.27
soil cont1	120	5.627	0.8063	14.33
soil cont2	104	12.216	1.5785	12.92

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLK	0	0.000	0.0000

DATE 87/03/17

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: BARIUM TEST CODE: BAUT SAMPLE TYPE: SOIL
UNIT: Vegetation/Soil/Sediment SUPERVISOR: L. Pastorek

METHOD CODE:535BA0 - HMPNSOIL - ICP/AES
REVISION NO: Original DATE: 1980
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- Approximately 20 g
Container- Glass jars (4 oz) with bakelite screw cap.
Preservative- None
Other- Samples are air-dried and ground to less than 45 mesh.

SAMPLE PREPARATION:Partial Extn.-Yes Total Extn.- % Extracted- ND
Procedure- Weigh 0.500g sample into a calibrated digestion tube and add 1.5 ml HNO3 . Mix and stand at room temperature for 2 hrs. Add 4.5 ml HCl to each tube, mix and stand at room temperature for another hour. Digest in a hot block for 1 hour at 40°C, 1 hour at 60°C, 1 hour at 80°C and 4 hrs at 100°C allowing 1/2 hr. ramping times
NOTE: The final volume should be less than 2 ml; if more, continue heating at 125°C to accomplish this.
Dilute with DDW to 25 ml and mix thoroughly.
Settle out suspended particles overnight and decant clear supernatant into disposable plastic tubes for analysis by AES-ICP. Samples requiring 5 or more metals are analyzed by AES-ICP (see below).
INTERFERENCES: Several, which are compensated for by the computer program.
REPORTING RESULTS: Two significant figures. (µg/g Ba)
INSTRUMENTATION: For the determination of 5 or more elements:
Inductively coupled Plasma Emission Spectrometer- Jobin-Yvon 1 m.JY48P with Autosampler & DEC computer.PET interface to LIS.(see "Remarks").
Calibration Range: 0 - 10 mg/l.
Resolution: Four significant figures (0.0001 mg/l).
Sensitivity: Not applicable (NA).
Instrument Detection Limit:.002 mg/l.
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.1 to 70 ug/g.
Accuracy- Not determined (ND).
Precision of Controls-

	A	B
mean	84 µg/g	
std. dev.	4.2 µg/g	
R.S.D.	5.1 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	0.27	0.86	3.5
mean	4.6	28	48

W 0.5 µg/g T 2.5 µg/g

CONTROL LIMITS: µg/g

		W.L. (x ±2σ)	R.L. (x ±3σ)
Control	L.L.	76	71
QCRS85-1	U.L.	92	97

REMARKS:- Determination of boron MUST be on Atom Scan 2400 only.
- Extraction % (±) based on average recovery of 4 samples from Soil Research Institute - SO1, SO2, SO3 & SO4.
- Soils rich in organic matter should be ashed (like vegetation) prior to digestion.
- Samples requiring only Mg, Al and/or Ca are analyzed by Atom Scan.

SUMMARY REPORT OF QUALITY CONTROL DATA

BARIUM IN SOIL

Operating Range =0.1000to 70.0 ug/g

IN-RUN DUPLICATES

Range	<0.1000	0.1000to14.00	14.00 to35.00	35.00 to70.0	>70.0
no.	1	2	12	27	26
s.w.		1.0352	0.8573	3.4849	
mean		2.8039	28.2724	48.2005	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
QCRS85-1	132	83.594	4.2384	5.07

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
rb	65	0.002	0.0023

DATE 87/03/31

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: BERYLLIUM TEST CODE: BEUT SAMPLE TYPE: SOIL
UNIT: Vegetation/Soil/Sediment SUPERVISOR: L. Pastorek

METHOD CODE:535BA0 - HMPNSOIL - ICP/AES
REVISION NO: Original DATE: 1980
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- Approximately 20 g
Container- Glass jars (4 oz) with bakelite screw cap.
Preservative- None
Other- Samples are air-dried and ground to less than 45 mesh.

SAMPLE PREPARATION:Partial Extn.-Yes Total Extn.- % Extracted- ND
Procedure- Weigh 0.500g sample into a calibrated digestion tube and add 1.5 ml HNO_3 . Mix and stand at room temperature for 2 hrs. Add 4.5 ml HCl to each tube, mix and stand at room temperature for another hour. Digest in a hot block for 1 hour at 40°C, 1 hour at 60°C, 1 hour at 80°C and 4 hrs at 100°C allowing 1/2 hr. ramping times
NOTE: The final volume should be less than 2 ml; if more, continue heating at 125°C to accomplish this.
Dilute with DDW to 25 ml and mix thoroughly.
Settle out suspended particles overnight and decant clear supernatant into disposable plastic tubes for analysis by AES-ICP. Samples requiring 5 or more metals are analyzed by AES-ICP (see below).
INTERFERENCES: Several, which are compensated for by the computer program.
REPORTING RESULTS: Two significant figures. (µg/g Be)
INSTRUMENTATION: For the determination of 5 or more elements:
Inductively coupled Plasma Emission Spectrometer- Jobin-Yvon 1 m.JY48P with Autosampler & DEC computer.PET interface to LIS.(see "Remarks").
Calibration Range: 0 - 10 mg/l.
Resolution: Four significant figures (0.0001 mg/l).
Sensitivity: Not applicable (NA).
Instrument Detection Limit: .003 mg/l.
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.2 to 650 µg/g.
Accuracy- Not determined (ND).
Precision of Controls-

	A	B
mean	1.2 µg/g	
std. dev.	0.085µg/g	
R.S.D.	7.6 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	ND	ND	ND
mean	ND	ND	ND

W 0.5 µg/g T 2.5 µg/g

CONTROL LIMITS:

Control	µg/g	W.L. (x ±2σ)	R.L. (x ±3σ)
QCRS85-1	L.L.	1.0	0.94
	U.L.	1.4	1.5

REMARKS:- Determination of boron MUST be on Atom Scan 2400 only.
- Extraction % (±) based on average recovery of 4 samples from Soil Research Institute - SO1, SO2, SO3 & SO4.
- Soils rich in organic matter should be ashed (like vegetation) prior to digestion.
- Samples requiring only Mg, Al and/or Ca are analyzed by Atom Scan.

SUMMARY REPORT OF QUALITY CONTROL DATA

BERYLLIUM IN SOIL

Operating Range =0.2000to 10.0 ug/g

IN-RUN DUPLICATES

Range	<0.2000	0.2000to2.00	2.00 to5.00	5.00 to10.0	>10.0
no.	61	7	0	0	0
s.w.		0.3921	0.0000	0.0000	
mean		1.0252	0.0000	0.0000	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
QCRS85-1	5	1.123	0.0849	7.56

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
rb	49	0.001	0.0005

DATE 87/03/31

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: BORON TEST CODE: BBUT SAMPLE TYPE: SOIL
UNIT: Vegetation/Soil/Sediment SUPERVISOR: L. Pastorek

METHOD CODE:535BA0 - ICP/AES -HMPNSOIL (AS)
REVISION NO: Original DATE: 1980
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- Approximately 20 g
Container- Glass jars (4 oz) with bakelite screw cap.
Preservative- None
Other- Samples are air-dried and ground to less than 45 mesh.

SAMPLE PREPARATION:Partial Extn.-Yes Total Extn.- % Extracted-ND
Procedure- Weigh 0.500g sample into a calibrated digestion tube and add 1.5 ml HNO3 . Mix and stand at room temperature for 2 hrs. Add 4.5 ml HCl to each tube, mix and stand at room temperature for another hour. Digest in a hot block for 1 hour at 40°C, 1 hour at 60°C 1 hour at 80°C and 4 hrs at 100°C allowing 1/2 hr. ramping times
NOTE: The final volume should be less than 2 ml; if more, continue heating at 125°C to accomplish this.
Dilute with DDW to 25 ml and mix thoroughly.
Settle out suspended particles overnight and decant clear supernatant into disposable plastic tubes for analysis by AES-ICP. Samples requiring Ca, Mg, Al are analyzed by the Atomscan 2400.
INTERFERENCES: Several, which are compensated for by the computer program.
REPORTING RESULTS: Two significant figures. µg/g B
INSTRUMENTATION:
Inductively coupled Plasma Emission Spectrometer- Atom Scan 2400 with autosampler & computer.PET interface to LIS.(See "Remarks").
Calibration Range: 0 -10 mg/l.
Resolution: Four significant figures (0.0001 mg/l).
Sensitivity: Not applicable (NA).
Instrument Detection Limit:0.05 mg/l.
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 1.0 to 25 µg/g.
Accuracy- Not determined (ND).
Precision of Controls- A B

mean	3.7 µg/g
std. dev.	1.0 µg/g
R.S.D.	28 %

Precision of Duplicates-

	low range	mid range	high range
s.d.	0.76	0.77	ND
mean	4.4	6.5	ND

W 1 µg/g T 5 µg/g

CONTROL LIMITS: µg/g W.L. (x ±2σ) R.L. (x ±3σ)

Control	L.L.	1.7	0.70
QCRS85-1	U.L.	5.7	6.7

REMARKS:- Determination of boron MUST be on Atom Scan 2400 only.
- Extraction % (±) based on average recovery of 4 samples from Soil Research Institute - S01, S02, S03 & S04.
- Samples requiring only B, Mg, Al and/or Ca are analyzed by Atom Scan

SUMMARY REPORT OF QUALITY CONTROL DATA

BORON-AS IN SOIL

Operating Range =1.0000to 25.0 ug/g

IN-RUN DUPLICATES

Range	<1.0000	1.0000to5.00	5.00 to12.50	12.50 to25.0	>25.0
no.	0	2	1	0	0
s.w.		0.7551	0.7700	0.0000	
mean		4.4111	6.5026	0.0000	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
S85-1	6	3.706	1.0347	27.92

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BL	0	0.000	0.0000

DATE 87/04/22

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: CADMIUM TEST CODE: CDUT SAMPLE TYPE: SOIL
UNIT: Vegetation/Soil/Sediment SUPERVISOR: L. Pastorek

METHOD CODE:535BA0 - HMPNSOIL - ICP/AES
REVISION NO: Original DATE: 1980
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- Approximately 20 g
Container- Glass jars (4 oz) with bakelite screw cap.
Preservative- None
Other- Samples are air-dried and ground to less than 45 mesh.

SAMPLE PREPARATION:Partial Extn.-Yes Total Extn.- % Extracted-79±9
Procedure- Weigh 0.500g sample into a calibrated digestion tube and add 1.5 ml HNO3 . Mix and stand at room temperature for 2 hrs. Add 4.5 ml HCl to each tube, mix and stand at room temperature for another hour. Digest in a hot block for 1 hour at 40°C, 1 hour at 60°C, 1 hour at 80°C and 4 hrs at 100°C allowing 1/2 hr. ramping times
NOTE: The final volume should be less than 2 ml; if more, continue heating at 125°C to accomplish this.
Dilute with DDW to 25 ml and mix thoroughly.
Settle out suspended particles overnight and decant clear supernatant into disposable plastic tubes for analysis by AES-ICP. Samples requiring 5 or more metals are analyzed by AES-ICP (see below).
INTERFERENCES: Several, which are compensated for by the computer program.
REPORTING RESULTS: Two significant figures. (µg/g of Cd)
INSTRUMENTATION: For the determination of 5 or more elements:
Inductively coupled Plasma Emission Spectrometer - Jobin-Yvon 1 m. JY48P with Autosampler & DEC computer.PET interface to LIS.("Remarks")
Calibration Range: 0 - 10 mg/l.
Resolution: Four significant figures (0.0001 mg/l).
Sensitivity: Not applicable (NA).
Instrument Detection Limit: .002 mg/l.
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.05 to 5 µg/g.
Accuracy- Not determined (ND).
Precision of Controls-

	A	B
mean	13 µg/g	
std. dev.	0.97 µg/g	
R.S.D.	7.7 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	0.13	0.12	
mean	0.49	1.8	

W .05 µg/g T .25 µg/g

CONTROL LIMITS: µg/g

Control		W.L. (x ±2σ)	R.L. (x ±3σ)
QCRS85-1	L.L.	11	10
	U.L.	15	16

REMARKS:- Determination of boron MUST be on Atom Scan 2400 only.
- Extraction % (±) based on average recovery of 4 samples from Soil Research Institute - S01, S02, S03 & S04.
- Soils rich in organic matter should be ashed (like vegetation) prior to digestion.
- Samples requiring only Mg, Al and/or Ca are analyzed by Atom Scan.

SUMMARY REPORT OF QUALITY CONTROL DATA

CADMIUM IN SOIL

Operating Range =0.0500to 5.0 ug/g

IN-RUN DUPLICATES

Range	<0.0500	0.0500to1.00	1.00 to2.50	2.50 to5.0	>5.0
no.	34	21	4	0	9
s.w.		0.1564	0.1204	0.0000	
mean		0.3703	1.7514	0.0000	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
QCRS85-1	132	12.606	0.9655	7.66

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
rb	60	0.001	0.0013

DATE 87/03/31

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: CADMIUM TEST CODE: CDUT SAMPLE TYPE: SOIL
UNIT: Vegetation/Soil/Sediment SUPERVISOR: L. Pastorek

METHOD CODE:535BA0 - HMPNSOIL - AAS
REVISION NO: Original DATE: 1980
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- Approximately 20 g
Container- Glass jars (4 oz) with Bakelite screw cap.
Preservative- None
Other- Samples are air-dried and ground to less than 45 mesh.

SAMPLE PREPARATION:Partial Extn.-Yes Total Extn.- % Extracted-79±9
Procedure- Weigh 0.500g sample into a calibrated digestion tube and add 1.5 ml HNO_3 . Mix and stand at room temperature for 2 hrs. Add 4.5 ml HCl to each tube, mix and stand at room temperature for another hour. Digest in a hot block for 1 hour at 40°C, 1 hour at 60°C 1 hour at 80°C and 4 hrs at 100°C allowing 1/2 hr. ramping times NOTE: The final volume should be less than 2 ml; if more, continue heating at 125°C to accomplish this.
Add 0.5 ml 5% KCl, dilute with DDW to 25 ml and mix thoroughly. Settle out suspended particles overnight and decant clear supernatant into disposable plastic tubes for analysis. Samples requiring the determination of < 5 elements and/or Na, K or Ag are analyzed by AAS.
INTERFERENCES: Several, which may be compensated for by background correction.
REPORTING RESULTS: Two significant figures. (µg/g Cd)
INSTRUMENTATION: PE 5000 atomic absorption spectrophotometer, PE Autosampler 50 and a PE Automatic Burner Controller,interfaced with Commodore Pet 4032, Tractor Printer 4022P & CBM 805C dual drive.
Calibration Range: 0 - 0.500 mg/l.
Resolution: 0.001 mg/l.
Sensitivity: conc. for absorbance of approx. 0.20 units (1.5 mg/l)
Instrument Detection Limit: 0.004 mg/l.
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.25 to 25 µg/g.
Accuracy- Not determined (ND).
Precision of Controls-

	A	B
mean	14 µg/g	
std. dev.	0.80 µg/g	
R.S.D.	5.9 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	0.044		
mean	0.74		

W .2 µg/g T 1.0 µg/g

CONTROL LIMITS:

µg/g	W.L. (x ±2σ)	R.L. (x ±3σ)
L.L.	12	12
U.L.	16	16

REMARKS: Control QCRS85-1
Extraction % (±) based on average recovery of 4 samples from Soil Research Institute - S01, S02, S03 & S04.

SUMMARY REPORT OF QUALITY CONTROL DATA

CADMIUM-AA IN SOIL

Operating Range =0.2000to 25.0 ug/g

IN-RUN DUPLICATES

Range	<0.2000	0.2000to5.00	5.00 to12.50	12.50 to25.0	>25.0
no.	0	8	0	0	0
s.w.		0.0440	0.0000	0.0000	
mean		0.7350	0.0000	0.0000	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
S85-1	17	13.470	0.8000	5.94

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.

DATE 87/04/23

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: CALCIUM TEST CODE: CAUT SAMPLE TYPE: SOIL
UNIT: Vegetation/Soil/Sediment SUPERVISOR: L. Pastorek

METHOD CODE:535BA0 - HMPNSOIL - ICP/AES (AS)
REVISION NO: Original DATE: 1980
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- Approximately 20 g
Container- Glass jars (4 oz) with bakelite screw cap.
Preservative- None
Other- Samples are air-dried and ground to less than 45 mesh.

SAMPLE PREPARATION:Partial Extn.-Yes Total Extn.- % Extracted-45±15
Procedure- Weigh 0.500g sample into a calibrated digestion tube and add 1.5 ml HNO3 . Mix and stand at room temperature for 2 hrs. Add 4.5 ml HCl to each tube, mix and stand at room temperature for another hour. Digest in a hot block for 1 hour at 40°C, 1 hour at 60°C 1 hour at 80°C and 4 hrs at 100°C allowing 1/2 hr. ramping times
NOTE: The final volume should be less than 2 ml; if more, continue heating at 125°C to accomplish this.
Dilute with DDW to 25 ml and mix thoroughly.
Settle out suspended particles overnight and decant clear supernatant into disposable plastic tubes for analysis by AES-ICP. Samples requiring 5 or more metals are analyzed by AES-ICP (see below).
INTERFERENCES: Several, which are compensated for by the computer program.
REPORTING RESULTS: Two significant figures. (µg/g Ca)
INSTRUMENTATION: For the determination of Ca, Mg, Al and/or B Inductively coupled Plasma Emission Spectrometer- Atom Scan 2400 with autosampler and computer.PET interface to LIS.(See "Remarks").
Calibration Range: 0 -2000 mg/l.
Resolution: Four significant figures (0.0001 mg/l).
Sensitivity: Not applicable (NA).
Instrument Detection Limit:4.0 mg/l.
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 10 to 20000 µg/g.
Accuracy- Not determined (ND).
Precision of Controls-

	A	B
mean	28000µg/g	
std. dev.	800 µg/g	
R.S.D.	2.8 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	70	740	460
mean	1500	8500	14000

W 20 µg/g T 100 µg/g

CONTROL LIMITS: µg/g

		W.L. (x ±2σ)	R.L. (x±3σ)
Control	L.L.	26000	26000
QCRS85-1	U.L.	30000	30000

REMARKS:- Determination of boron MUST be on Atom Scan 2400 only.
- Extraction % (±) based on average recovery of 4 samples from Soil Research Institute - SO1, SO2, SO3 & SO4.
- Samples requiring Ca can also be analyzed by Jobin-Yvon.

SUMMARY REPORT OF QUALITY CONTROL DATA

CALCIUM-AS IN SOIL

Operating Range =10.000to 20000. ug/g

IN-RUN DUPLICATES

Range	<10.000	10.000to4000.0	4000.0to10000.	10000.to20000.	>20000.
no.	0	1	2	7	0
s.w.		70.1240	741.2500	456.7000	
mean		1457.780	8477.300	13695.00	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
RS85-1	9	28234.03	799.9900	2.83

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BL	900	-21.73	0.7306

DATE 87/04/22

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: CALCIUM TEST CODE: CAUT SAMPLE TYPE: SOIL
UNIT: Vegetation/Soil/Sediment SUPERVISOR: L. Pastorek

METHOD CODE:535BA0 - HMPNSOIL - ICP/AES (JY)
REVISION NO: Original DATE: 1980
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- Approximately 20 g
Container- Glass jars (4 oz) with bakelite screw cap.
Preservative- None
Other- Samples are air-dried and ground to less than 45 mesh.

SAMPLE PREPARATION:Partial Extn.-Yes Total Extn.- % Extracted-45±15
Procedure- Weigh 0.500g sample into a calibrated digestion tube and add 1.5 ml HNO_3 . Mix and stand at room temperature for 2 hrs. Add 4.5 ml HCl to each tube, mix and stand at room temperature for another hour. Digest in a hot block for 1 hour at 40°C, 1 hour at 60°C 1 hour at 80°C and 4 hrs at 100°C allowing 1/2 hr. ramping times
NOTE: The final volume should be less than 2 ml; if more, continue heating at 125°C to accomplish this.
Dilute with DDW to 25 ml and mix thoroughly.
Settle out suspended particles overnight and decant clear supernatant into disposable plastic tubes for analysis by AES-ICP. Samples requiring 5 or more metals are analyzed by AES-ICP (see below).
INTERFERENCES: Several, which are compensated for by the computer program.
REPORTING RESULTS: Two significant figures. (µg/g Ca)
INSTRUMENTATION: For the determination of 5 or more elements:
Inductively coupled Plasma Emission Spectrometer- Jobin-Yvon 1 m.JY48P with Autosampler & DEC computer.PET interface to LIS.(See "Remarks").
Calibration Range: 0 -1500 mg/l.
Resolution: Four significant figures (0.0001 mg/l).
Sensitivity: Not applicable (NA).
Instrument Detection Limit:0.173 mg/l.
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 10 to 20000 µg/g.
Accuracy- Not determined (ND).
Precision of Controls-

	A	B
mean	28000µg/g	
std. dev.	1200 µg/g	
R.S.D.	4.2 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	140	280	290
mean	2200	6800	12000

W 20 µg/g T 100 µg/g

CONTROL LIMITS: µg/g

Control		W.L. (x ±2σ)	R.L. (x ±3σ)
QCRS85-1	L.L.	26000	24000
	U.L.	30000	32000

REMARKS:- Determination of boron MUST be on Atom Scan 2400 only.
- Extraction % (±) based on average recovery of 4 samples from Soil Research Institute - SO1, SO2, SO3 & SO4.
- Samples requiring only Mg, Al, Ca and/or B are analyzed by Atom Scan Precision of controls for Atom Scan listed under "B".

SUMMARY REPORT OF QUALITY CONTROL DATA

CALCIUM-jy IN SOIL

Operating Range =10.000to 20000. ug/g

IN-RUN DUPLICATES

Range	<10.000	10.000to4000.0	4000.0to10000.	10000.to20000.	>20000.
no.	2	28	11	14	13
s.w.		137.8620	280.1960	287.5820	
mean		2205.370	6783.700	12530.57	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
QCRS85-1	132	27522.91	1147.730	4.17

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
rb	61	0.112	0.0810

DATE 87/03/31

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: CHROMIUM TEST CODE: CRUT SAMPLE TYPE: SOIL
UNIT: Vegetation/Soil/Sediment SUPERVISOR: L. Pastorek

METHOD CODE:535BA0 - HMPNSOIL - ICP/AES
REVISION NO: Original DATE: 1980
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- Approximately 20 g
Container- Glass jars (4 oz) with bakelite screw cap.
Preservative- None
Other- Samples are air-dried and ground to less than 45 mesh.

SAMPLE PREPARATION:Partial Extn.-Yes Total Extn.- % Extracted-56+20
Procedure- Weigh 0.500g sample into a calibrated digestion tube and add 1.5 ml HNO_3 . Mix and stand at room temperature for 2 hrs. Add 4.5 ml HCl to each tube, mix and stand at room temperature for another hour. Digest in a hot block for 1 hour at 40C, 1 hour at 60C, 1 hour at 80C and 4 hrs at 100C (allowing 1/2 hr. ramping times)
NOTE: The final volume should be less than 2 ml; if more, continue heating at 125C to accomplish this.
Dilute with DDW to 25 ml and mix thoroughly.
Settle out suspended particles overnight and decant clear supernatant into disposable plastic tubes for analysis by AES-ICP. Samples requiring 5 or more metals are analyzed by AES-ICP (see below).
INTERFERENCES: Several, which are compensated for by the computer program.
REPORTING RESULTS: Two significant figures. (µg/g Cr)
INSTRUMENTATION: For the determination of 5 or more elements:
Inductively coupled Plasma Emission Spectrometer - Jobin-Yvon 1 m. JY48P with Autosampler & DEC computer.PET interface to LIS.("Remarks")
Calibration Range: 0 - 10 mg/l.
Resolution: Four significant figures (0.0001 mg/l).
Sensitivity: Not applicable (NA).
Instrument Detection Limit: .007 mg/l.
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.4 to 50 µg/g.
Accuracy- Not determined (ND).
Precision of Controls-

	A	B
mean	230 µg/g	
std. dev.	16 µg/g	
R.S.D.	6.9 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	0.41	1.3	1.3
mean	6.8	18	34

W 1 µg/g T 5 µg/g

CONTROL LIMITS: µg/g

Control		W.L. (x ±2σ)	R.L. (x ±3σ)
QCRS85-1	L.L.	200	180
	U.L.	260	280

REMARKS:- Determination of boron MUST be on Atom Scan 2400 only.
- Extraction % (±) based on average recovery of 4 samples from Soil Research Institute - SO1, SO2, SO3 & SO4.
- Soils rich in organic matter should be ashed (like vegetation) prior to digestion.
- Samples requiring only Mg, Al and/or Ca are analyzed by Atom Scan.

SUMMARY REPORT OF QUALITY CONTROL DATA

CHROMIUM IN SOIL

Operating Range =0.4000to 50.0 ug/g

IN-RUN DUPLICATES

Range	<0.4000	0.4000to10.00	10.00 to25.00	25.00 to50.0	>50.0
no.	1	2	21	27	17
s.w.		0.4120	1.3303	1.2720	
mean		6.8302	18.5451	34.3344	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
QCRS85-1	131	229.551	15.8507	6.91

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
rb	76	0.013	0.0180

DATE 87/03/31

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: COBALT TEST CODE: COUT SAMPLE TYPE: SOIL
UNIT: Vegetation/Soil/Sediment SUPERVISOR: L. Pastorek

METHOD CODE:535BA0 - HMPNSOIL - ICP/AES
REVISION NO: Original DATE: 1980
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- Approximately 20 g
Container- Glass jars (4 oz) with bakelite screw cap.
Preservative- None
Other- Samples are air-dried and ground to less than 45 mesh.

SAMPLE PREPARATION:Partial Extn.-Yes Total Extn.- % Extracted-43±15
Procedure- Weigh 0.500g sample into a calibrated digestion tube and add 1.5 ml HNO_3 . Mix and stand at room temperature for 2 hrs. Add 4.5 ml HCl to each tube, mix and stand at room temperature for another hour. Digest in a hot block for 1 hour at 40°C, 1 hour at 60°C, 1 hour at 80°C and 4 hrs at 100°C allowing 1/2 hr. ramping times
NOTE: The final volume should be less than 2 ml; if more, continue heating at 125°C to accomplish this.
Dilute with DDW to 25 ml and mix thoroughly.
Settle out suspended particles overnight and decant clear supernatant into disposable plastic tubes for analysis by AES-ICP. Samples requiring 5 or more metals are analyzed by AES-ICP (see below).
INTERFERENCES: Several, which are compensated for by the computer program.
REPORTING RESULTS: Two significant figures. (µg/g Co)
INSTRUMENTATION: For the determination of 5 or more elements:
Inductively coupled Plasma Emission Spectrometer - Jobin-Yvon 1 m. JY48P with Autosampler & DEC computer.PET interface to LIS.("Remarks")
Calibration Range: 0 - 10 mg/l.
Resolution: Four significant figures (0.0001 mg/l).
Sensitivity: Not applicable (NA).
Instrument Detection Limit: .002 mg/l.
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.1 to 25 µg/g.
Accuracy- Not determined (ND).
Precision of Controls-

	A	B
mean	36 µg/g	
std. dev.	2.3 µg/g	
R.S.D.	6.3 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	0.066	0.30	0.69
mean	3.2	9.5	16

W 0.2 µg/g T 1.0 µg/g

CONTROL LIMITS: µg/g

Control		W.L. (x ±2σ)	R.L. (x ±3σ)
QCRS85-1	L.L.	31	29
	U.L.	41	43

REMARKS:- Determination of boron MUST be on Atom Scan 2400 only.
- Extraction % (±) based on average recovery of 4 samples from Soil Research Institute - S01, S02, S03 & S04.
- Soils rich in organic matter should be ashed (like vegetation) prior to digestion.
- Samples requiring only Mg, Al and/or Ca are analyzed by Atom Scan.

SUMMARY REPORT OF QUALITY CONTROL DATA

COBALT IN SOIL

Operating Range =0.1000to 25.0 ug/g

IN-RUN DUPLICATES

Range	<0.1000	0.1000to5.00	5.00 to12.50	12.50 to25.0	>25.0
no.	2	2	40	18	6
s.w.		0.0658	0.3049	0.6890	
mean		3.2296	9.4861	16.1332	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
QCRS85-1	127	36.475	2.2976	6.30

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
rb	55	0.002	0.0027

DATE 87/03/31

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: COPPER TEST CODE: CUUT SAMPLE TYPE: SOIL
UNIT: Vegetation/Soil/Sediment SUPERVISOR: L. Pastorek

METHOD CODE:535BA0 - HMPNSOIL - ICP/AES
REVISION NO: Original DATE: 1980
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- Approximately 20 g
Container- Glass jars (4 oz) with bakelite screw cap.
Preservative- None
Other- Samples are air-dried and ground to less than 45 mesh.

SAMPLE PREPARATION:Partial Extn.-Yes Total Extn.- % Extracted-70±7
Procedure- Weigh 0.500g sample into a calibrated digestion tube and add 1.5 ml HNO_3 . Mix and stand at room temperature for 2 hrs. Add 4.5 ml HCl to each tube, mix and stand at room temperature for another hour. Digest in a hot block for 1 hour at 40°C, 1 hour at 60°C 1 hour at 80°C and 4 hrs at 100°C allowing 1/2 hr. ramping times
NOTE: The final volume should be less than 2 ml; if more, continue heating at 125°C to accomplish this.
Dilute with DDW to 25 ml and mix thoroughly.
Settle out suspended particles overnight and decant clear supernatant into disposable plastic tubes for analysis by AES-ICP. Samples requiring 5 or more metals are analyzed by AES-ICP (see below).
INTERFERENCES: Several, which are compensated for by the computer program.
REPORTING RESULTS: Two significant figures. (µg/g Cu)
INSTRUMENTATION: For the determination of 5 or more elements:
Inductively coupled Plasma Emission Spectrometer - Jobin-Yvon 1 m. Y48P with Autosampler & DEC computer. PET interface to LIS.("Remarks")
Calibration Range: 0 - 10 mg/l.
Resolution: Four significant figures (0.0001 mg/l).
Sensitivity: Not applicable (NA).
Instrument Detection Limit:.006 mg/l.
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.3 to 100 ug/g.
Accuracy- Not determined (ND).
Precision of Controls-

	A	B
mean	1600ug/g	
std. dev.	63 ug/g	
R.S.D.	4.0%	

Precision of Duplicates-

	low range	mid range	high range
s.d.	sd 1.6	2.6	5.2
mean	ug/g 14	30	73

W 1 µg/g T 5 µg/g

CONTROL LIMITS: µg/g

Control		W.L. (x ±2σ)	R.L. (x ±3σ)
QCRS85-1	L.L.	1500	1400
	U.L.	1700	1800

REMARKS:- Determination of boron MUST be on Atom Scan 2400 only.
- Extraction % (±) based on average recovery of 4 samples from Soil Research Institute - S01, S02, S03 & S04.
- Soils rich in organic matter should be ashed (like vegetation) prior to digestion.
- Samples requiring only Mg, Al and/or Ca are analyzed by Atom Scan.

SUMMARY REPORT OF QUALITY CONTROL DATA

COPPER IN SOIL

Operating Range =0.3000to 100.0 ug/g

IN-RUN DUPLICATES

Range	<0.3000	0.3000to20.00	20.00 to50.00	50.00 to100.0	>100.0
no.	0	13	20	8	27
s.w.		1.6101	2.5680	5.1997	
mean		14.4416	29.8548	72.7087	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
QCRS85-1	132	1594.339	63.3716	3.97

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
rb	91	0.021	0.0287

DATE 87/03/31

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: COPPER TEST CODE: CUUT SAMPLE TYPE: SOIL
UNIT: Vegetation/Soil/Sediment SUPERVISOR: L. Pastorek

METHOD CODE:535BA0 - HMPNSOIL - AAS
REVISION NO: Original DATE: 1980
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- Approximately 20 g
Container- Glass jars (4 oz) with bakelite screw cap.
Preservative- None
Other- Samples are air-dried and ground to less than 45 mesh.

SAMPLE PREPARATION:Partial Extn.-Yes Total Extn.- % Extracted-70±7
Procedure- Weigh 0.500g sample into a calibrated digestion tube and add 1.5 ml HNO_3 . Mix and stand at room temperature for 2 hrs. Add 4.5 ml HCl to each tube, mix and stand at room temperature for another hour. Digest in a hot block for 1 hour at 40°C, 1 hour at 60°C 1 hour at 80°C and 4 hrs at 100°C allowing 1/2 hr. ramping times
NOTE: The final volume should be less than 2 ml; if more, continue heating at 125°C to accomplish this.
Add 0.5 ml 5% KCl, dilute with DDW to 25 ml and mix thoroughly. Settle out suspended particles overnight and decant clear supernatant into disposable plastic tubes for analysis. Samples requiring the determination of < 5 elements and/or Na, K or Ag are analyzed by AAS.
INTERFERENCES: Several, which may be compensated for by background correction.
REPORTING RESULTS: Two significant figures. (µg/g Cu)
INSTRUMENTATION: PE 5000 atomic absorption spectrophotometer, PE Autosampler 50 and a PE Automatic Burner Controller,interfaced with Commodore Pet 4032, Tractor Printer 4022P & CBM 805C dual drive.
Calibration Range: 0 - 5 mg/l.
Resolution: 0.01 mg/l.
Sensitivity: conc. for absorbance of approx. 0.20 units (4.0 mg/l)
Instrument Detection Limit: 0.02 mg/l.
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 1.0 to 250 µg/g.
Accuracy- Not determined (ND).
Precision of Controls-

	A	B
mean	1600 µg/g	
std. dev.	41 µg/g	
R.S.D.	2.5 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	1.0	ND	ND
mean	13		

W 1 µg/g T 5 µg/g

CONTROL LIMITS:

µg/g	W.L. (x ±2σ)	R.L. (x ±3σ)
L.L.	1500	1500
U.L.	1700	1700

REMARKS: Control QCRS85-1
Extraction % (±) based on average recovery of 4 samples from Soil Research Institute - S01, S02, S03 & S04.

SUMMARY REPORT OF QUALITY CONTROL DATA

COPPER-AA IN SOIL

Operating Range =1.0000to 250.0 ug/g

IN-RUN DUPLICATES

Range	<1.0000	1.0000to50.00	50.00 to125.00	125.00to250.0	>250.0
no.	0	2	0	0	0
s.w.		0.1000	0.0000	0.0000	
mean		13.0500	0.0000	0.0000	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
S85-1	6	1616.670	40.8250	2.53

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.

DATE 87/04/23

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: IRON TEST CODE: FEUT SAMPLE TYPE: SOIL
UNIT: Vegetation/Soil/Sediment SUPERVISOR: L. Pastorek

METHOD CODE:535BA0 - HMPNSOIL - ICP/AES
REVISION NO: Original DATE: 1980
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- Approximately 20 g
Container- Glass jars (4 oz) with bakelite screw cap.
Preservative- None
Other- Samples are air-dried and ground to less than 45 mesh.

SAMPLE PREPARATION:Partial Extn.-Yes Total Extn.- % Extracted-66±13
Procedure- Weigh 0.500g sample into a calibrated digestion tube and add 1.5 ml HNO3 . Mix and stand at room temperature for 2 hrs. Add 4.5 ml HCl to each tube, mix and stand at room temperature for another hour. Digest in a hot block for 1 hour at 40±C, 1 hour at 60°C, 1 hour at 80°C and 4 hrs at 100°C allowing 1/2 hr. ramping times
NOTE: The final volume should be less than 2 ml; if more, continue heating at 125°C to accomplish this.
Dilute with DDW to 25 ml and mix thoroughly.
Settle out suspended particles overnight and decant clear supernatant into disposable plastic tubes for analysis by AES-ICP. Samples requiring 5 or more metals are analyzed by AES-ICP (see below).
INTERFERENCES: Several, which are compensated for by the computer program.
REPORTING RESULTS: Two significant figures. (µg/g Fe)
INSTRUMENTATION: For the determination of 5 or more elements:
Inductively coupled Plasma Emission Spectrometer- Jobin-Yvon 1 m.JY48P with Autosampler & DEC computer.PET interface to LIS.(see "Remarks").
Calibration Range: 0 - 1000 mg/l.
Resolution: Four significant figures (0.0001 mg/l).
Sensitivity: Not applicable (NA).
Instrument Detection Limit:.02 mg/l.
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 1 to 25000 ug/g.
Accuracy- Not determined (ND).
Precision of Controls-

	A	B
mean	27000ug/g	
std. dev.	1000 ug/g	
R.S.D.	3.7 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.		320	620
mean		10000	17000

W 200 µg/g T 1000 µg/g

CONTROL LIMITS: µg/g

		W.L. (x ±2σ)	R.L. (x ±3σ)
Control	L.L.	25000	24000
QCRS85-1	U.L.	29000	30000

REMARKS:- Determination of boron MUST be on Atom Scan 2400 only.
- Extraction % (±) based on average recovery of 4 samples from Soil Research Institute - SO1, SO2, SO3 & SO4.
- Soils rich in organic matter should be ashed (like vegetation) prior to digestion.
- Samples requiring only Mg, Al and/or Ca are analyzed by Atom Scan.

SUMMARY REPORT OF QUALITY CONTROL DATA

IRON IN SOIL

Operating Range =1.0000to 25000. ug/g

IN-RUN DUPLICATES

Range	<1.0000	1.0000to5000.0	5000.0to12500.	12500.to25000.	>25000.
no.	1	0	15	42	10
s.w.		0.0000	322.7120	621.5140	
mean		0.0000	10319.66	16729.73	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
QCRS85-1	132	27100.06	1010.168	3.73

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
rb	43	0.026	0.1028

DATE 87/03/31

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: LEAD TEST CODE: PBUT SAMPLE TYPE: SOIL
UNIT: Vegetation/Soil/Sediment SUPERVISOR: L. Pastorek

METHOD CODE:535BA0 -HMPNSOIL - ICP/AES
REVISION NO: Original DATE: 1980
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- Approximately 20 g
Container- Glass jars (4 oz) with bakelite screw cap.
Preservative- None
Other- Samples are air-dried and ground to less than 45 mesh.

SAMPLE PREPARATION:Partial Extn.-Yes Total Extn.- % Extracted-85±15
Procedure- Weigh 0.500g sample into a calibrated digestion tube and add 1.5 ml HNO3 . Mix and stand at room temperature for 2 hrs. Add 4.5 ml HCl to each tube, mix and stand at room temperature for another hour. Digest in a hot block for 1 hour at 40±C, 1 hour at 60°C, 1 hour at 80°C and 4 hrs at 100°C allowing 1/2 hr. ramping times
NOTE: The final volume should be less than 2 ml; if more, continue heating at 125°C to accomplish this.
Dilute with DDW to 25 ml and mix thoroughly.
Settle out suspended particles overnight and decant clear supernatant into disposable plastic tubes for analysis by AES-ICP. Samples requiring 5 or more metals are analyzed by AES-ICP (see below).
INTERFERENCES: Several, which are compensated for by thee computer program.
REPORTING RESULTS: Two significant figures. (µg/g Pb)
INSTRUMENTATION: For the determination of 5 or more elements:
Inductively coupled Plasma Emission Spectrometer- Jobin-Yvon 1 m.JY48P with Autosampler & DEC computer.PET interface to LIS.(see "Remarks").
Calibration Range: 0 - 10 mg/l.
Resolution: Four significant figures (0.0001 mg/l).
Sensitivity: Not applicable (NA).
Instrument Detection Limit:.02 mg/l.
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 1 to 100 ug/g.
Accuracy- Not determined (ND).
Precision of Controls-

	A	B
mean	950 ug/g	
std. dev.	32 ug/g	
R.S.D.	3.3 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	1.9	1.3	6.1
mean	11	33	73

W 2 µg/g T 10 µg/g

CONTROL LIMITS: µg/g

		W.L. (x ±2σ)	R.L. (x ±3σ)
Control	L.L.	890	850
QCRS85-1	U.L.	1000	1000

REMARKS:- Determination of boron MUST be on Atom Scan 2400 only.
- Extraction % (±) based on average recovery of 4 samples from Soil Research Institute - SO1, SO2, SO3 & SO4.
- Soils rich in organic matter should be ashed (like vegetation) prior to digestion.
- Samples requiring only Mg, Al and/or Ca are analyzed by Atom Scan.

SUMMARY REPORT OF QUALITY CONTROL DATA

LEAD IN SOIL

Operating Range =1.0000to 100.0 ug/g

IN-RUN DUPLICATES

Range	<1.0000	1.0000to20.00	20.00 to50.00	50.00 to100.0	>100.0
no.	1	24	19	8	16
s.w.		1.9328	1.2805	6.1307	
mean		11.4177	32.8229	72.8474	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
QCRS85-1	132	952.763	31.7323	3.33

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
rb	76	0.017	0.0184

DATE 87/03/31

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: LEAD TEST CODE: PBUT SAMPLE TYPE: SOIL
UNIT: Vegetation/Soil/Sediment SUPERVISOR: L. Pastorek

METHOD CODE:535BA0 HMPNSOIL - AAS
REVISION NO: Original DATE: 1980
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- Approximately 20 g
Container- Glass jars (4 oz) with bakelite screw cap.
Preservative- None
Other- Samples are air-dried and ground to less than 45 mesh.

SAMPLE PREPARATION:Partial Extn.-Yes Total Extn.- % Extracted-85+15
Procedure- Weigh 0.500g sample into a calibrated digestion tube and add 1.5 ml HNO_3 . Mix and stand at room temperature for 2 hrs. Add 4.5 ml HCl to each tube, mix and stand at room temperature for another hour. Digest in a hot block for 1 hour at 40C, 1 hour at 60C, 1 hour at 80C and 4 hrs at 100C (allowing 1/2 hr. ramping times)
NOTE: The final volume should be less than 2 ml; if more, continue heating at 125C to accomplish this. Add 0.5 ml of 5 % KCl, dilute with DDW to 25 ml and mix thoroughly.
Settle out suspended particles overnight and decant clear supernatant into disposable plastic tubes for analysis. Samples requiring < 5 metals are analyzed by AAS. (see below).
INTERFERENCES: Several, which are compensated for by background correction.
REPORTING RESULTS: Two significant figures. (µg/g Pb)
INSTRUMENTATION:For the determination of <5 elements and/or Na, K & Ag a PE 5000 atomic absorption spectrophotometer(AAS),PE Autosampler, PE Automatic Burner Controller, and computer is used
Calibration Range: 0 - 20 mg/l.
Resolution: 0.01 mg/l.
Sensitivity:Conc. for approx. 0.20 absorbance units: 20 mg/l.
Instrument Detection Limit: 0.06 mg/l.
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 2.0 to 1000 µg/g.
Accuracy- Not determined (ND).
Precision of Controls-

	A	B
mean	1100 µg/g	
std. dev.	77 µg/g	
R.S.D.	7.3 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	3.8	27	29
mean	65	360	760

W 2 µg/g T 10 µg/g

CONTROL LIMITS: µg/g

Control		W.L. (x ±2σ)	R.L. (x±3σ)
QCRS85-1	L.L.	950	870
	U.L.	1200	1300

REMARKS:
- Extraction % (±) based on average recovery of 4 samples from Soil Research Institute - S01, S02, S03 & S04.
- Soils rich in organic matter should be ashed (like vegetation) prior to digestion.

SUMMARY REPORT OF QUALITY CONTROL DATA

LEAD-AA IN SOIL

Operating Range =2.0000to 1000.0 ug/g

IN-RUN DUPLICATES

Range	<2.0000	2.0000to200.00	200.00to500.00	500.00to1000.	>1000.0
no.	0	5	10	11	17
s.w.		3.8079	27.3861	29.4649	
mean		65.1000	364.0000	758.6364	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
qcrs85-1	76	1058.421	76.8037	7.26

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.

DATE 87/04/23

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: MAGNESIUM TEST CODE: MGUT SAMPLE TYPE: SOIL
UNIT: Vegetation/Soil/Sediment SUPERVISOR: L. Pastorek

METHOD CODE:535BA0 - HMPNSOIL - ICP/AES (JY)
REVISION NO: Original DATE: 1980
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- Approximately 20 g
Container-Glass jars (4 oz.) with Bakelite screw caps.
Preservative- None
Other- Samples are air-dried and ground to less than 45 mesh.

SAMPLE PREPARATION:Partial Extn.-Yes Total Extn.- % Extracted-86±25
Procedure- Weigh 0.500g sample into a calibrated digestion tube and add 1.5 ml HNO3 . Mix and stand at room temperature for 2 hrs. Add 4.5 ml HCl to each tube, mix and stand at room temperature for another hour. Digest in a hot block for 1 hour at 40°C, 1 hour at 60°C 1 hour at 80°C and 4 hrs at 100°C allowing 1/2 hr. ramping times
NOTE: The final volume should be less than 2 ml; if more, continue heating at 125°C to accomplish this.
Dilute with DDW to 25 ml and mix thoroughly.
Settle out suspended particles overnight and decant clear supernatant into disposable plastic tubes for analysis by AES-ICP. Samples requiring 5 or more metals are analyzed by AES-ICP (see below).
INTERFERENCES: Several, which are compensated for by the computer program.
REPORTING RESULTS: Two significant figures. (µg/g Mg)
INSTRUMENTATION: For the determination of 5 or more elements:
Inductively coupled Plasma Emission Spectrometer- Jobin-Yvon 1 m.JY48P with Autosampler & DEC computer.PET interface to LIS.(See "Remarks").
Calibration Range: 0 -50 mg/l.
Resolution: Four significant figures (0.0001 mg/l).
Sensitivity: Not applicable (NA).
Instrument Detection Limit:0.164 mg/l.
PERFORMANCE CHARACTERISTICS:
Routine Operating Range-8 to 10000 µg/g.
Accuracy- Not determined (ND).
Precision of Controls-

	A	B
mean	5600µg/g	
std. dev.	220 µg/g	
R.S.D.	4.0 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	83	140	160
mean	1500	3100	6900

W 10 µg/g T 50 µg/g

CONTROL LIMITS: µg/g

Control		W.L. (x ±2σ)	R.L. (x ±3σ)
QCRS85-1	L.L.	5100	4900
	U.L.	6100	6300

REMARKS:- Determination of boron MUST be on Atom Scan 2400 only.
- Extraction % (±) based on average recovery of 4 samples from Soil Research Institute - S01, S02, S03 & S04.
- Samples requiring only Mg, Al and/or Ca are analyzed by Atom Scan.

SUMMARY REPORT OF QUALITY CONTROL DATA

MAGNESIUM IN SOIL

Operating Range =8.0000to 10000. ug/g

IN-RUN DUPLICATES

Range	<8.0000	8.0000to2000.0	2000.0to5000.0	5000.0to10000.	>10000.
no.	0	10	44	8	6
s.w.		83.0180	145.7700	158.0400	
mean		1492.260	3060.470	6916.940	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
QCRS85-1	132	5617.420	225.5840	4.02

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
rb	38	0.059	0.0437

DATE 87/03/31

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: MAGNESIUM TEST CODE: MGUT SAMPLE TYPE: SOIL
UNIT: Vegetation/Soil/Sediment SUPERVISOR: L. Pastorek

METHOD CODE:535BA0 - ICP/AES -HMPNSOIL (AS)
REVISION NO: Original DATE: 1980
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- Approximately 20 g
Container- Glass jars (4 oz) with bakelite screw cap.
Preservative- None
Other- Samples are air-dried and ground to less than 45 mesh.

SAMPLE PREPARATION:Partial Extn.-Yes Total Extn.- % Extracted-86±25
Procedure- Weigh 0.500g sample into a calibrated digestion tube and add 1.5 ml HNO3 . Mix and stand at room temperature for 2 hrs. Add 4.5 ml HCl to each tube, mix and stand at room temperature for another hour. Digest in a hot block for 1 hour at 40°C, 1 hour at 60°C 1 hour at 80°C and 4 hrs at 100°C allowing 1/2 hr. ramping times
NOTE: The final volume should be less than 2 ml; if more, continue heating at 125°C to accomplish this.
Dilute with DDW to 25 ml and mix thoroughly.
Settle out suspended particles overnight and decant clear supernatant into disposable plastic tubes for analysis by AES-ICP. Samples requiring 5 or more metals are analyzed by AES-ICP (see below).
INTERFERENCES: Several, which are compensated for by the computer program.
REPORTING RESULTS: Two significant figures. (µg/g Mg)
INSTRUMENTATION: For the determination of Ca, Mg, Al, and/or B only. Inductively coupled Plasma Emission Spectrometer- Atom Scan 2400 with autosampler and computer.PET interface to LIS.(See "Remarks").
Calibration Range: 0 -50 mg/l.
Resolution: Four significant figures (0.0001 mg/l).
Sensitivity: Not applicable (NA).
Instrument Detection Limit:2.0 mg/l.
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 10 to 10000 µg/g.
Accuracy- Not determined (ND).
Precision of Controls-

	A	B
mean	6300 µg/g	
std. dev.	160 µg/g	
R.S.D.	2.5 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	27	ND	ND
mean	2000	ND	ND

W 10 µg/g T 50 µg/g

CONTROL LIMITS: µg/g

Control		W.L. (x ±2σ)	R.L. (x ±3σ)
QCRS85-1	L.L.	6000	5800
	U.L.	6300	6500

REMARKS:- Determination of boron MUST be on Atom Scan 2400 only.
- Extraction % (±) based on average recovery of 4 samples from Soil Research Institute - SO1, SO2, SO3 & SO4.
- Samples requiring Mg can also be analyzed by Jobin-Yvon.

SUMMARY REPORT OF QUALITY CONTROL DATA

MAGNESIUM-AS IN SOIL

Operating Range =10.000to 10000. ug/g

IN-RUN DUPLICATES

Range	<10.000	10.000to2000.0	2000.0to5000.0	5000.0to10000.	>10000.
no.	0	1	0	0	0
s.w.		26.6860	0.0000	0.0000	
mean		1965.500	0.0000	0.0000	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
RS85-1	4	6319.540	159.5350	2.52

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BL	200	-1.360	0.0498

DATE 87/04/22

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: MANGANESE TEST CODE: MNUT SAMPLE TYPE: SOIL
UNIT: Vegetation/Soil/Sediment SUPERVISOR: L. Pastorek

METHOD CODE:535BA0 -HMPNSOIL - ICP/AES
REVISION NO: Original DATE: 1980
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- Approximately 20 g
Container- Glass jars (4 oz) with bakelite screw cap.
Preservative- None
Other- Samples are air-dried and ground to less than 45 mesh.

SAMPLE PREPARATION:Partial Extn.-Yes Total Extn.- % Extracted-69±15
Procedure- Weigh 0.500g sample into a calibrated digestion tube and add 1.5 ml HNO3 . Mix and stand at room temperature for 2 hrs. Add 4.5 ml HCl to each tube, mix and stand at room temperature for another hour. Digest in a hot block for 1 hour at 40±C, 1 hour at 60°C, 1 hour at 80°C and 4 hrs at 100°C allowing 1/2 hr. ramping times
NOTE: The final volume should be less than 2 ml; if more, continue heating at 125°C to accomplish this.
Dilute with DDW to 25 ml and mix thoroughly.
Settle out suspended particles overnight and decant clear supernatant into disposable plastic tubes for analysis by AES-ICP. Samples requiring 5 or more metals are analyzed by AES-ICP (see below).
INTERFERENCES: Several, which are compensated for by the computer program.
REPORTING RESULTS: Two significant figures. (µg/g Mn)
INSTRUMENTATION: For the determination of 5 or more elements:
Inductively coupled Plasma Emission Spectrometer- Jobin-Yvon 1 m.JY48P with Autosampler & DEC computer.PET interface to LIS.(see "Remarks").
Calibration Range: 0 - 10 mg/l.
Resolution: Four significant figures (0.0001 mg/l).
Sensitivity: Not applicable (NA).
Instrument Detection Limit: 0.008 mg/l.
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.4 to 500 µg/g.
Accuracy- Not determined (ND).
Precision of Controls-

	A	B
mean	400 µg/g	
std. dev.	16 µg/g	
R.S.D.	4.0 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	4.8	7.5	8.5
mean	66	190	320

W 5 µg/g T 25 µg/g

CONTROL LIMITS: µg/g

		W.L. (x ±2σ)	R.L. (x ±3σ)
Control	L.L.	370	350
QCRS85-1	U.L.	430	450

REMARKS:- Determination of boron MUST be on Atom Scan 2400 only.
- Extraction % (±) based on average recovery of 4 samples from Soil Research Institute - S01, S02, S03 & S04.
- Soils rich in organic matter should be ashed (like vegetation) prior to digestion.
- Samples requiring only Mg, Al and/or Ca are analyzed by Atom Scan.

SUMMARY REPORT OF QUALITY CONTROL DATA

MANGANESE IN SOIL

Operating Range =0.4000to 500.0 ug/g

IN-RUN DUPLICATES

Range	<0.4000	0.4000to100.00	100.00to250.00	250.00to500.0	>500.0
no.	1	8	25	29	5
s.w.		4.7564	7.5134	8.4884	
mean		65.9565	191.5284	321.4681	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
QCRS85-1	132	397.198	15.8358	3.99

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
rb	39	0.003	0.0018

DATE 87/03/31

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME:Mercury TEST CODE:HGUT SAMPLE TYPE:Soil
UNIT: Biomaterials SUPERVISOR: R. S. Sadana

METHOD CODE:541AF1 TYPE: Flameless AAS
REVISION NO: Original DATE: May, 1984
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- Approximately 20 g.
Container- Glass jars (4 oz.) with bakelite screw caps.
Preservative- None
Other- Samples are air-dried and ground to less than 45 mesh.

SAMPLE PREPARATION:Partial Extn.- Total Extn.-Yes % Extracted-
Procedure- Weigh approx. 0.250 g of sample into a 50 ml Folin-Wu digestion tube. Add 5 ml of acid mixture (4:1 -H_2SO_4:HNO_3) and place the tube in a Technicon aluminum hot block (1½ h @ 150°C;1½ h @ 190°;2 h @ 250°C).
Cool overnight,then dilute to 25 ml with distilled water.
Run in batches of about 28 samples.

Treat blanks and calibration standards in exactly the same manner.
Determine mercury in the entire volume. The measurement step is automated and is based on the evolution of atomic vapour of mercury (wavelength - 254nm) by the addition of a reducing agent.
INTERFERENCES: Water vapour; organic solvents.
Very high concentration of cations.
REPORTING RESULTS:Two significant figures (ug/g Hg).
INSTRUMENTATION: Automated sampler and peristaltic pump (Technicon or Gilson). Laboratory Data Control U.V. monitor (Pharmacia).
Calibration Range: 0 - 16.0 ng/ml
Resolution: 0.025 ng/ml (one division on recorder chart paper)
Sensitivity:1.0 ng/ml reads 0.4 absorbance (20 divs on chart)
Instrument Detection Limit: 0.1 ng/ml
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.01 - 4.0 µg/g
Accuracy- 100% at 1.1 ug/g
Precision of Controls-

	A	B
mean	.46	
std. dev.	.034	
R.S.D.	7.5%	

Precision of Duplicates-

	low range	mid range	high range
s.d.	.018		
mean	.17		

W 0.01 µg/g T 0.05 µg/g

CONTROL LIMITS: µg/g	W.L.(x ±2σ)	R.L.(x ±3σ)
L.L.	0.39	0.36
U.L.	0.53	0.56

REMARKS:

- Precision based on CCIW round robin.
- Detection Limit - 2x std. dev. of low range within-run duplicates.
- Accuracy - Ratio of mean and cert. value in ref. mat. (%).

SUMMARY REPORT OF QUALITY CONTROL DATA

MERCURY IN SOIL

Operating Range =0.0100to 4.0 ug/g

IN-RUN DUPLICATES

Range	<0.0100	0.0100to0.80	0.80 to2.00	2.00 to4.0	>4.0
no.	2	18	0	1	1
s.w.		0.0183	0.0000	0.1768	
mean		0.1690	0.0000	2.4450	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
con 684	104	0.454	0.0347	7.64

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.

DATE 87/03/30

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: MOLYBDENUM TEST CODE: MOUT SAMPLE TYPE: SOIL
UNIT: Vegetation/Soil/Sediment SUPERVISOR: L. Pastorek

METHOD CODE:535BA0 - HMPNSOIL - ICP/AES
REVISION NO: Original DATE: 1980
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- Approximately 20 g
Container- Glass jars (4 oz) with bakelite screw cap.
Preservative- None
Other- Samples are air-dried and ground to less than 45 mesh.

SAMPLE PREPARATION:Partial Extn.-Yes Total Extn.- % Extracted- ND
Procedure- Weigh 0.500g sample into a calibrated digestion tube and add 1.5 ml HNO_3 . Mix and stand at room temperature for 2 hrs. Add 4.5 ml HCl to each tube, mix and stand at room temperature for another hour. Digest in a hot block for 1 hour at 40°C, 1 hour at 60°C, 1 hour at 80°C and 4 hrs at 100°C allowing 1/2 hr. ramping times
NOTE: The final volume should be less than 2 ml; if more, continue heating at 125°C to accomplish this.
Dilute with DDW to 25 ml and mix thoroughly.
Settle out suspended particles overnight and decant clear supernatant into disposable plastic tubes for analysis by AES-ICP. Samples requiring 5 or more metals are analyzed by AES-ICP (see below).
INTERFERENCES: Several, which are compensated for by the computer program.
REPORTING RESULTS: Two significant figures. (µg/g Mo)
INSTRUMENTATION: For the determination of 5 or more elements:
Inductively coupled Plasma Emission Spectrometer- Jobin-Yvon 1 m.JY48P with Autosampler & DEC computer.PET interface to LIS.(see "Remarks").
Calibration Range: 0 - 10 mg/l.
Resolution: Four significant figures (0.0001 mg/l).
Sensitivity: Not applicable (NA).
Instrument Detection Limit:.004 mg/l.
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.2 to 10 µg/g.
Accuracy- Not determined (ND).
Precision of Controls-

	A	B
mean	5.9 µg/g	
std. dev.	0.79 µg/g	
R.S.D.	13 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	0.29	3.8	0.0099
mean	1.4	2.7	7.8

W 0.2 µg/g T 1.0 µg/g

CONTROL LIMITS: µg/g

Control		W.L. (x ±2σ)	R.L. (x ±3σ)
QCRS85-1	L.L.	4.3	3.5
	U.L.	7.5	8.3

REMARKS:- Determination of boron MUST be on Atom Scan 2400 only.
- Extraction % (±) based on average recovery of 4 samples from Soil Research Institute - SO1, SO2, SO3 & SO4.
- Soils rich in organic matter should be ashed (like vegetation) prior to digestion.
- Samples requiring only Mg, Al and/or Ca are analyzed by Atom Scan.

SUMMARY REPORT OF QUALITY CONTROL DATA

MOLYBDENUM IN SOIL

Operating Range =0.2000to 10.0 ug/g

IN-RUN DUPLICATES

Range	<0.2000	0.2000to2.00	2.00 to5.00	5.00 to10.0	>10.0
no.	58	8	1	1	0
s.w.		0.4921	3.8317	0.0099	
mean		1.2109	2.7094	7.7957	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
QCRS85-1	127	5.920	0.7870	13.29

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
rb	70	0.006	0.0053

DATE 87/03/31

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: NICKEL TEST CODE: NIUT SAMPLE TYPE: SOIL
UNIT: Vegetation/Soil/Sediment SUPERVISOR: L. Pastorek

METHOD CODE:535BA0 - HMPNSOIL - ICP/AES
REVISION NO: Original DATE: 1980
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- Approximately 20 g
Container- Glass jars (4 oz) with bakelite screw cap.
Preservative- None
Other- Samples are air-dried and ground to less than 45 mesh.

SAMPLE PREPARATION:Partial Extn.-Yes Total Extn.- % Extracted-69±7
Procedure- Weigh 0.500g sample into a calibrated digestion tube and add 1.5 ml HNO3 . Mix and stand at room temperature for 2 hrs. Add 4.5 ml HCl to each tube, mix and stand at room temperature for another hour. Digest in a hot block for 1 hour at 40°C, 1 hour at 60°C, 1 hour at 80°C and 4 hrs at 100°C allowing 1/2 hr. ramping times
NOTE: The final volume should be less than 2 ml; if more, continue heating at 125°C to accomplish this.
Dilute with DDW to 25 ml and mix thoroughly.
Settle out suspended particles overnight and decant clear supernatant into disposable plastic tubes for analysis by AES-ICP. Samples requiring 5 or more metals are analyzed by AES-ICP (see below).
INTERFERENCES: Several, which are compensated for by the computer program.
REPORTING RESULTS: Two significant figures. (µg/g of Ni)
INSTRUMENTATION: For the determination of 5 or more elements:
Inductively coupled Plasma Emission Spectrometer- Jobin-Yvon 1 m.JY48P with Autosampler & DEC computer.PET interface to LIS.(see "Remarks").
Calibration Range: 0 - 10 mg/l.
Resolution: Four significant figures (0.0001 mg/l).
Sensitivity: Not applicable (NA).
Instrument Detection Limit: .004 mg/l.
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.2 to 100 µg/g.
Accuracy- Not determined (ND).
Precision of Controls-

	A	B
mean	640µg/g	
std. dev.	25 µg/g	
R.S.D.	3.9%	

Precision of Duplicates-

	low range	mid range	high range
s.d.	.64	1.6	2.9
mean	10	27	72

W 0.5 µg/g T 2.5 µg/g

CONTROL LIMITS: µg/g

Control		W.L. (x ±2σ)	R.L. (x ±3σ)
QCRS85-1	L.L.	590	560
	U.L.	670	720

REMARKS:- Determination of boron MUST be on Atom Scan 2400 only.
- Extraction % (±) based on average recovery of 4 samples from Soil Research Institute - SO1, SO2, SO3 & SO4.
- Soils rich in organic matter should be ashed (like vegetation) prior to digestion.
- Samples requiring only Mg, Al and/or Ca are analyzed by Atom Scan.

SUMMARY REPORT OF QUALITY CONTROL DATA

NICKEL IN SOIL

Operating Range =0.2000to 100.0 ug/g

IN-RUN DUPLICATES

Range	<0.2000	0.2000to20.00	20.00 to50.00	50.00 to100.0	>100.0
no.	2	26	18	7	15
s.w.		0.6427	1.6527	2.8670	
mean		10.0771	26.8005	72.1536	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
QCRS85-1	130	637.973	24.6463	3.86

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
rb	60	0.004	0.0039

DATE 87/03/31

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: NICKEL TEST CODE: NIUT SAMPLE TYPE: SOIL
UNIT: Vegetation/Soil/Sediment SUPERVISOR: L. Pastorek

METHOD CODE:535BA0 -HMPNSOIL - AAS
REVISION NO: Original DATE: 1980
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- Approximately 20 g
Container- Glass jars (4 oz) with bakelite screw cap.
Preservative- None
Other- Samples are air-dried and ground to less than 45 mesh.

SAMPLE PREPARATION:Partial Extn.-Yes Total Extn.- % Extracted-69±7
Procedure- Weigh 0.500g sample into a calibrated digestion tube and add 1.5 ml HNO3 . Mix and stand at room temperature for 2 hrs. Add 4.5 ml HCl to each tube, mix and stand at room temperature for another hour. Digest in a hot block for 1 hour at 40°C, 1 hour at 60°C 1 hour at 80°C and 4 hrs at 100°C allowing 1/2 hr. ramping times
NOTE: The final volume should be less than 2 ml; if more, continue heating at 125°C to accomplish this.
Add 0.5 ml 5% KCl, dilute with DDW to 25 ml and mix thoroughly. Settle out suspended particles overnight and decant clear supernatant into disposable plastic tubes for analysis. Samples requiring the determination of < 5 elements and/or Na, K or Ag are analyzed by AAS.
INTERFERENCES: Several, which may be compensated for by background correction.
REPORTING RESULTS: Two significant figures. (µg/g Ni)
INSTRUMENTATION: PE 5000 atomic absorption spectrophotometer, PE Autosampler 50 and a PE Automatic Burner Controller,interfaced with Commodore Pet 4032, Tractor Printer 4022P & CBM 805C dual drive.
Calibration Range: 0 - 5 mg/l.
Resolution: 0.01 mg/l.
Sensitivity: conc. for absorbance of approx. 0.20 units (7.0 mg/l)
Instrument Detection Limit: 0.04 mg/l.
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.5 to 250 µg/g.
Accuracy- Not determined (ND).
Precision of Controls-

	A	B
mean	740 µg/g	
std. dev.	27 µg/g	
R.S.D.	3.7 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	1.0	ND	ND
mean	10.0		

W .5 µg/g T 2.0 µg/g

CONTROL LIMITS:

µg/g	W.L. (x ±2σ)	R.L. (x ±3σ)
L.L.	690	660
U.L.	790	820

REMARKS: Control QCRS85-1
Extraction % (±) based on average recovery of 4 samples from Soil Research Institute - S01, S02, S03 & S04.

SUMMARY REPORT OF QUALITY CONTROL DATA

NICKEL-AA IN SOIL

Operating Range =0.5000to 250.0 ug/g

IN-RUN DUPLICATES

Range	<0.5000	0.5000to50.00	50.00 to125.00	125.00to250.0	>250.0
no.	0	1	0	0	0
s.w.		0.0000	0.0000	0.0000	
mean		10.0000	0.0000	0.0000	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
S85-1	5	736.000	27.0190	3.67

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.

DATE 87/04/23

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: POTASSIUM TEST CODE: KKUT SAMPLE TYPE: SOIL
UNIT: Vegetation/Soil/Sediment SUPERVISOR: L. Pastorek

METHOD CODE:535BE1 - AAS -HMPNSOIL
REVISION NO: Original DATE: 1980
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- Approximately 20 g
Container- Glass jar (4 oz) with bakelite screw cap.
Preservative- None
Other- Samples are air-dried and ground to less than 45 mesh.

SAMPLE PREPARATION:Partial Extn.-Yes Total Extn.- % Extracted-
Procedure- Weigh 0.500g sample into a calibrated digestion tube and add 1.5 ml HNO3 . Mix and stand at room temperature for 2 hrs. Add 4.5 ml HCl to each tube, mix and stand at room temperature for another hour. Digest in a hot block for 1 hour at 40°C, 1 hour at 60°C 1 hour at 80°C and 4 hrs at 100°C allowing 1/2 hr. ramping times
NOTE: The final volume should be less than 2 ml; if more, continue heating at 125°C to accomplish this.
Add 0.5 ml 5% CsCl, dilute with DDW to 25 ml and mix thoroughly. Settle out suspended particles overnight and decant clear supernatant into disposable plastic tubes for analysis. Samples requiring the determination of < 5 elements and/or Na, K or Ag are analyzed by AAS.
INTERFERENCES: 1000 ppm Cs is added to help prevent partial ionization.
REPORTING RESULTS: Two significant figures. (µg/g K)
INSTRUMENTATION: PE 5000 AAS (in emission mode.)
PE Autosampler 50 and a PE Automatic Burner Controller,interfaced with Commodore Pet 4032, Tractor Printer 4022P & CBM 805C dual drive.
Calibration Range: 0 - 20 mg/l.
Resolution: 0.01 mg/l.
Sensitivity: conc. for absorbance of approx. 0.2 units (2.0 mg/l)
Instrument Detection Limit: 0.2 mg/l.
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 5 to 1000 µg/g.
Accuracy- Not determined (ND).
Precision of Controls-

		A	B
	mean	1600	
	std. dev.	270	
	R.S.D.	17 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	ND	7.4	46
mean	ND	340	890

W 5 µg/g T 25 µg/g

CONTROL LIMITS: ug/g

	W.L. (x ±2σ)	R.L. (x ±3σ)
L.L.	1100	790
U.L.	2100	2400

REMARKS: Control QCRS85-1
Extraction % (±) ND -based on average recovery of 4 samples from Soil Research Institute - S01, S02, S03 & S04.

SUMMARY REPORT OF QUALITY CONTROL DATA

POTASSIUM IN SOIL

Operating Range =5.0000to 1000.0 ug/g

IN-RUN DUPLICATES

Range	<5.0000	5.0000to200.00	200.00to500.00	500.00to1000.	>1000.0
no.	0	0	9	12	3
s.w.		0.0000	7.4536	45.7803	
mean		0.0000	343.3333	893.7500	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
qcrs85-1	30	1623.333	268.6920	16.55

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.

DATE 87/04/03

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME:Selenium TEST CODE:SEUT SAMPLE TYPE:Soil
UNIT:Biomaterials SUPERVISOR: R. S. Sadana

METHOD CODE:542AF3 TYPE: Semi-aut. hydr. gen - flameless AAS
REVISION NO: Original DATE: January, 1983
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- Approximately 2 g
Container- Glass jar with bakelite screw cap
Preservative- None
Other-

SAMPLE PREPARATION:Partial Extn.- Total Extn.-Yes % Extracted->90
Procedure- Weigh 60 mg (45 mesh) sample into a 18 x 150 mm pyrex graduated test tube. Add 3 ml of acid mixture (6 nitric: 3 sulphuric: 1 perchloric). Process in batches of 80 samples including blanks, calibration standards and controls.
Digest in an aluminum hot block at a medium setting on the hot plate for 14 hrs until dense white fumes appear.
Cool, add 0.5 ml of distilled water, then add 2 ml conc. HCl.
Dilute to 15 ml with distilled water and mix.
Feed the prepared solutions to the automated system for the determination of selenium by the hydride-FAAS technique.
INTERFERENCES: Excessive concentrations of Cu, Fe and Ni may interfere.
REPORTING RESULTS:2 dec. places <10, 1 dec. <100, 0 dec. >100 µg/g
INSTRUMENTATION: Atomic absorption spectrophotometer (Varian Techtron 1200 & AA-5, with chart recorder, peristaltic pump and an autosampler.
Open ended heated quartz "T" cell (0.6x10cm); gas-liquid separator.
Calibration Range:0 - 40 ng/ml (linear <20 ng/ml)
Resolution: 0.01 absorbance (unexpanded scale)
Sensitivity:0.02 µg/ml reads 0.20 abs.
Instrument Detection Limit: 0.001 µg/ml
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.3 to 10 µg/g
Accuracy- 99% (NBS orchard leaves)
Precision of Controls-

	A	B
mean	.51	.70
std. dev.	.15	.24
R.S.D.	30	34

Precision of Duplicates-

	low range	mid range	high range
s.d.	.04	.43	
mean	.52	3.88	

W 0.2 µg/g T 1.0 µg/g

CONTROL LIMITS:

REMARKS:

- Detection Limit - 3x std. dev. of low range within-run duplicates.
- Accuracy - Ratio of mean to cert. value in ref. mat. X 100

SUMMARY REPORT OF QUALITY CONTROL DATA

SELENIUM IN SOIL

Operating Range =0.3000to 10.0 ug/g

IN-RUN DUPLICATES

Range	<0.3000	0.3000to2.00	2.00 to5.00	5.00 to10.0	>10.0
no.	30	26	4	0	0
s.w.		0.0472	0.4272	0.0000	
mean		0.6970	3.8750	0.0000	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
orch leave	31	0.082	0.0523	63.78
veg contro	49	3.741	0.4038	10.79
soil cont1	66	0.510	0.1506	29.53
soil cont2	68	0.699	0.2396	34.28

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLK	0	0.000	0.0000

DATE 87/03/17

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: SILVER TEST CODE: AGUT SAMPLE TYPE: SOIL
UNIT: Vegetation/Soil/Sediment SUPERVISOR: L. Pastorek

METHOD CODE:535BA0 - AAS -HMPNSOIL
REVISION NO: Original DATE: 1980
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- Approximately 20 g
Container- Glass jar (4 oz) with bakelite screw cap.
Preservative- None
Other- Samples are air-dried and ground to less than 45 mesh.

SAMPLE PREPARATION:Partial Extn.-Yes Total Extn.- % Extracted-ND
Procedure- Weigh 0.500g sample into a calibrated digestion tube and add 1 ml H_2SO_4 (conc.). Add 2.5 ml HNO_3 (conc) and mix well.

Digest in a hot block for 1 hour at 60C, and 4.5 hrs at 175°C (allowing 1 hr. ramping times).
NOTE: The final volume should be less than 2 ml; if more, continue to heat at 175°C to accomplish this. Samples MUST fume during digestion.
Cool, dilute with DDW to 25 ml and mix thoroughly.
Settle out suspended particles overnight and decant clear supernatant into disposable plastic tubes for analysis by AAS. For the determination of < 5 elements and/or Na, K or Ag use the PE-5000.
INTERFERENCES: Several, which may be compensated for by background correction.
REPORTING RESULTS: Two significant figures. µg/g Ag
INSTRUMENTATION: PE 5000 atomic absorption spectrophotometer, PE Autosampler 50 and a PE Automatic Burner Controller, interfaced with Commodore Pet 4032, Tractor Printer 4022P & CBM 805C dual drive.
Calibration Range: 0 - 0.5 mg/l.
Resolution: 0.001 mg/l.
Sensitivity: conc. for absorbance of approx. 0.20 units (2.5 mg/l)
Instrument Detection Limit: 0.005 mg/l.
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.1 to 25 µg/g.
Accuracy- Not determined (ND).
Precision of Controls-

	A	B
mean	0.98 µg/g	
std. dev.	0.15 µg/g	
R.S.D.	15 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	0.14	ND	ND
mean	1.1		

W .1 µg/g T .5 µg/g

CONTROL LIMITS: µg/g

	W.L. (x ±2σ)	R.L. (x ±3σ)
L.L.	0.68	0.53
U.L.	1.3	1.4

REMARKS: Control QCRS85-1
Extraction % (±) ND -based on average recovery of 4 samples from Soil Research Institute - SO1, SO2, SO3 & SO4.

SUMMARY REPORT OF QUALITY CONTROL DATA

SILVER-AA IN SOIL

Operating Range =0.1000to 25.0 ug/g

IN-RUN DUPLICATES

Range	<0.1000	0.1000to5.00	5.00 to12.50	12.50 to25.0	>25.0
no.	0	1	0	0	1
s.w.		0.1410	0.0000	0.0000	
mean		1.1000	0.0000	0.0000	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
S85-1	8	0.980	0.1490	15.20

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.

DATE 87/04/23

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: SODIUM TEST CODE: NAUT SAMPLE TYPE: SOIL
UNIT: Vegetation/Soil/Sediment SUPERVISOR: L. Pastorek

METHOD CODE:535BE1 - AAS -HMPNSOIL
REVISION NO: Original DATE: 1980
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- Approximately 20 g
Container- Glass jar (4 oz) with bakelite screw cap.
Preservative- None
Other- Samples are air-dried and ground to less than 45 mesh.

SAMPLE PREPARATION:Partial Extn.-Yes Total Extn.- % Extracted-
Procedure- Weigh 0.500g sample into a calibrated digestion tube and add 1.5 ml HNO3 . Mix and stand at room temperature for 2 hrs. Add 4.5 ml HCl to each tube, mix and stand at room temperature for another hour. Digest in a hot block for 1 hour at 40°C, 1 hour at 60°C 1 hour at 80°C and 4 hrs at 100°C allowing 1/2 hr. ramping times
NOTE: The final volume should be less than 2 ml; if more, continue heating at 125°C to accomplish this.
Add 0.5 ml 5% KCl, dilute with DDW to 25 ml and mix thoroughly. Settle out suspended particles overnight and decant clear supernatant into disposable plastic tubes for analysis. Samples requiring the determination of < 5 elements and/or Na, K or Ag are analyzed by AAS.
INTERFERENCES: 1000 ppm K or Cs is added to prevent partial ionization.
REPORTING RESULTS: Two significant figures.
INSTRUMENTATION: PE 5000 AAS (in emission mode.)
PE Autosampler 50 and a PE Automatic Burner Controller,interfaced with Commodore Pet 4032, Tractor Printer 4022P & CBM 805C dual drive.
Calibration Range: 0 - 20 mg/l.
Resolution: 0.01 mg/l.
Sensitivity: conc. for absorbance of approx. 0.20 units (0.5 mg/l)
Instrument Detection Limit: 0.1 mg/l.
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 5.0 to 1000 ug/g.
Accuracy- Not determined (ND).
Precision of Controls-

	A	B
mean	550	
std. dev.	50	
R.S.D.	9.1 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	9.2	12	ND
mean	120	240	ND

W 5 µg/g T 25 µg/g

CONTROL LIMITS:

µg/g	W.L. (x ±2σ)	R.L. (x±3σ)
L.L.	450	400
U.L.	650	700

REMARKS: Control QCRS85-1
Extraction % (±) ND -based on average recovery of 4 samples from Soil Research Institute - S01, S02, S03 & S04.

SUMMARY REPORT OF QUALITY CONTROL DATA

SODIUM IN SOIL

Operating Range =5.0000to 1000.0 ug/g

IN-RUN DUPLICATES

Range	<5.0000	5.0000to200.00	200.00to500.00	500.00to1000.	>1000.0
no.	0	10	3	0	1
s.w.		9.2195	12.9099	0.0000	
mean		123.5000	243.3333	0.0000	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
qcrs85-1	39	550.256	49.8148	9.05

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.

DATE 87/04/03

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: STRONTIUM TEST CODE: SRUT SAMPLE TYPE: SOIL
UNIT: Vegetation/Soil/Sediment SUPERVISOR: L. Pastorek

METHOD CODE:535BA0 - HMPNSOIL - ICP/AES
REVISION NO: Original DATE: 1980
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- Approximately 20 g
Container- Glass jars (4 oz) with bakelite screw cap.
Preservative- None
Other- Samples are air-dried and ground to less than 45 mesh.

SAMPLE PREPARATION:Partial Extn.-Yes Total Extn.- % Extracted- ND
Procedure- Weigh 0.500g sample into a calibrated digestion tube and add 1.5 ml HNO3 . Mix and stand at room temperature for 2 hrs. Add 4.5 ml HCl to each tube, mix and stand at room temperature for another hour. Digest in a hot block for 1 hour at 40°C, 1 hour at 60°C 1 hour at 80°C and 4 hrs at 100°C allowing 1/2 hr. ramping times
NOTE: The final volume should be less than 2 ml; if more, continue heating at 125°C to accomplish this.
Dilute with DDW to 25 ml and mix thoroughly.
Settle out suspended particles overnight and decant clear supernatant into disposable plastic tubes for analysis by AES-ICP. Samples requiring 5 or more metals are analyzed by AES-ICP (see below).
INTERFERENCES: Several, which are compensated for by the computer program.
REPORTING RESULTS: Two significant figures. (µg/g Sr)
INSTRUMENTATION: For the determination of 5 or more elements:
Inductively coupled Plasma Emission Spectrometer- Jobin-Yvon 1 m.JY48P with Autosampler & DEC computer.PET interface to LIS.(see "Remarks").
Calibration Range: 0 - 10 mg/l.
Resolution: Four significant figures (0.0001 mg/l).
Sensitivity: Not applicable (NA).
Instrument Detection Limit: .00095 mg/l.
PERFORMANCE CHARACTERISTICS:
Routine Operating Range-.05 to 50 µg/g.
Accuracy- Not determined (ND).
Precision of Controls-

	A	B
mean	57 µg/g	
std. dev.	2.1 µg/g	
R.S.D.	3.7 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	1.4	1.6	1.1
mean	8.2	16	34

W 1 µg/g T 5 µg/g

CONTROL LIMITS: µg/g

Control		W.L. (x ±2σ)	R.L. (x ±3σ)
QCRS85-1	L.L.	53	51
	U.L.	61	63

REMARKS:- Determination of boron MUST be on Atom Scan 2400 only.
- Extraction % (±) based on average recovery of 4 samples from Soil Research Institute - SO1, SO2, SO3 & SO4.
- Soils rich in organic matter should be ashed (like vegetation) prior to digestion.
- Samples requiring only Mg, Al, Ca and/or B are analyzed by Atom Scan

SUMMARY REPORT OF QUALITY CONTROL DATA

STRONTIUM IN SOIL

Operating Range =0.0500to 50.0 ug/g

IN-RUN DUPLICATES

Range	<0.0500	0.0500to10.00	10.00 to25.00	25.00 to50.0	>50.0
no.	1	2	22	32	11
s.w.		1.3944	1.5779	1.1283	
mean		8.2384	16.3713	33.9586	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
QCRS85-1	132	56.908	2.1201	3.73

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
rb	59	0.001	0.0009

DATE 87/03/31

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: URANIUM TEST CODE: UUUT SAMPLE TYPE:SOIL
UNIT: Vegetation/Soil/Sediment SUPERVISOR: L. Pastorek

METHOD CODE:520AE2
REVISION NO: 2 DATE: Feb., 1986
NATURE OF LAST REVISION:Detectn meth & prepn. from fluoro to ICP/MS.

SAMPLE HANDLING:
Quantity Required- Approximately 20 g
Container-Glass jars (4 oz) with bakelite/screw cap or PET container
Preservative- None
Other-Samples are air-dried and ground to less 45 mesh

SAMPLE PREPARATION:Partial Extn.-Yes Total Extn.- % Extracted-
Procedure- Ash 0.2 g at 500C for 6 hrs in a 100 ml beaker. Add 5 ml 16N HNO3 to cooled sample. Digest to dryness.
Add 5 ml 8N HNO3 and warm contents of beaker for several minutes.
Wash into 50 ml graduated centrifuge tube and make up to 50 ml.
Centrifuge and analyze supernatant by ICP/MS.

INTERFERENCES: High concentrations of alkali metals.

REPORTING RESULTS: µg/g U.
INSTRUMENTATION: Sciex Elan 250 inductively coupled plasma mass spectrometer.

Calibration Range: 0 to 1.0 mg/l.
Resolution: 0.0001 mg/l.
Sensitivity:0.100 mg/l - between 35000 and 85000 counts per second
Instrument Detection Limit: 0.0001 mg/l.

PERFORMANCE CHARACTERISTICS:
Routine Operating Range-.01 to 50 µg/g.
Accuracy- Not determined (ND).
Precision of Controls-

	A	B
mean	16 µg/g	24
std. dev.	2.3 µg/g	3.1
R.S.D.	14 %	13 %

Precision of Duplicates-

	low range	mid range	high range
s.d.	0.32	0.21	2.26
mean	0.86	20.07	34.42

W 0.2 µg/g T 1.0 µg/g

CONTROL LIMITS: µg/g

Control		W.L. (x ±2σ)	R.L. (x ±3σ)
	L.L.	11	9.1
	U.L.	21	23

REMARKS:- Control limits:
- > ± 3 sd (standard deviations) on digested control samples before rejection of run.
- Conversion from mg/l to ug/g: ug/g = mg/l x 250.

SUMMARY REPORT OF QUALITY CONTROL DATA

URANIUM IN SOIL

Operating Range =0.0100to 50.0 ug/g

IN-RUN DUPLICATES

Range	<0.0100	0.0100to10.00	10.00 to25.00	25.00 to50.0	>50.0
no.	0	7	2	2	0
s.w.		0.3195	0.2090	2.2607	
mean		0.8560	20.0740	34.4170	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
cons1	20	16.336	2.3097	14.14
cons2	20	24.219	3.0974	12.79

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLK	0	0.000	0.0000

DATE 87/03/31

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: VANADIUM TEST CODE: VVUT SAMPLE TYPE: SOIL
UNIT: Vegetation/Soil/Sediment SUPERVISOR: L. Pastorek

METHOD CODE:535BA0 - HMPNSOIL - ICP/AES
REVISION NO: Original DATE: 1980
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- Approximately 20 g
Container- Glass jars (4 oz) with bakelite screw cap.
Preservative- None
Other- Samples are air-dried and ground to less than 45 mesh.

SAMPLE PREPARATION:Partial Extn.-Yes Total Extn.- % Extracted-65±10
Procedure- Weigh 0.500g sample into a calibrated digestion tube and add 1.5 ml HNO3 . Mix and stand at room temperature for 2 hrs. Add 4.5 ml HCl to each tube, mix and stand at room temperature for another hour. Digest in a hot block for 1 hour at 40°C, 1 hour at 60°C, 1 hour at 80°C and 4 hrs at 100°C allowing 1/2 hr. ramping times
NOTE: The final volume should be less than 2 ml; if more, continue heating at 125°C to accomplish this.
Dilute with DDW to 25 ml and mix thoroughly.
Settle out suspended particles overnight and decant clear supernatant into disposable plastic tubes for analysis by AES-ICP. Samples requiring 5 or more metals are analyzed by AES-ICP (see below).
INTERFERENCES: Several, which are compensated for by the computer program.
REPORTING RESULTS: Two significant figures. (µg/g V)
INSTRUMENTATION: For the determination of 5 or more elements:
Inductively coupled Plasma Emission Spectrometer- Jobin-Yvon 1 m.JY48P with Autosampler & DEC computer.PET interface to LIS.(see "Remarks").
Calibration Range: 0 - 10 mg/l.
Resolution: Four significant figures (0.0001 mg/l).
Sensitivity: Not applicable (NA).
Instrument Detection Limit: .01 mg/l.
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.5 to 50 µg/g.
Accuracy- Not determined (ND).
Precision of Controls-

	A	B
mean	35 µg/g	
std. dev.	2.4 µg/g	
R.S.D.	6.8 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	0.56	1.2	1.6
mean	7.6	20	32

W 1 µg/g T 5 µg/g

CONTROL LIMITS: µg/g

Control		W.L. (x ±2σ)	R.L. (x ±3σ)
QCRS85-1	L.L.	30	28
	U.L.	40	42

REMARKS:- Determination of boron MUST be on Atom Scan 2400 only.
- Extraction % (±) based on average recovery of 4 samples from Soil Research Institute - SO1, SO2, SO3 & SO4.
- Soils rich in organic matter should be ashed (like vegetation) prior to digestion.
- Samples requiring only Mg, Al and/or Ca are analyzed by Atom Scan.

SUMMARY REPORT OF QUALITY CONTROL DATA

VANADIUM IN SOIL

Operating Range =0.5000to 50.0 ug/g

IN-RUN DUPLICATES

Range	<0.5000	0.5000to10.00	10.00 to25.00	25.00 to50.0	>50.0
no.	1	1	13	48	5
s.w.		0.5586	1.1975	1.5915	
mean		7.5795	19.5317	31.6166	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
QCRS85-1	132	34.935	2.3784	6.81

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
rb	45	0.006	0.0052

DATE 87/03/31

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: ZINC TEST CODE: ZNUT SAMPLE TYPE: SOIL
UNIT: Vegetation/Soil/Sediment SUPERVISOR: L. Pastorek

METHOD CODE:535BA0 - HMPNSOIL - ICP/AES
REVISION NO: Original DATE: 1980
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- Approximately 20 g
Container- Glass jars (4 oz) with bakelite screw cap.
Preservative- None
Other- Samples are air-dried and ground to less than 45 mesh.

SAMPLE PREPARATION:Partial Extn.-Yes Total Extn.- % Extracted-85±5
Procedure- Weigh 0.500g sample into a calibrated digestion tube and add 1.5 ml HNO_3 . Mix and stand at room temperature for 2 hrs. Add 4.5 ml HCl to each tube, mix and stand at room temperature for another hour. Digest in a hot block for 1 hour at 40°C, 1 hour at 60°C, 1 hour at 80°C and 4 hrs at 100°C allowing 1/2 hr. ramping times
NOTE: The final volume should be less than 2 ml; if more, continue heating at 125°C to accomplish this.
Dilute with DDW to 25 ml and mix thoroughly.
Settle out suspended particles overnight and decant clear supernatant into disposable plastic tubes for analysis by AES-ICP. Samples requiring 5 or more metals are analyzed by AES-ICP (see below).
INTERFERENCES: Several, which are compensated for by the computer program.
REPORTING RESULTS: Two significant figures. (µg/g Zn)
INSTRUMENTATION: For the determination of 5 or more elements:
Inductively coupled Plasma Emission Spectrometer - Jobin-Yvon 1 m. JY48P with Autosampler & DEC computer.PET interface to LIS.("Remarks")
Calibration Range: 0 - 10 mg/l.
Resolution: Four significant figures (0.0001 mg/l).
Sensitivity: Not applicable (NA).
Instrument Detection Limit: .02 mg/l.
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 1 to 100 µg/g.
Accuracy- Not determined (ND).
Precision of Controls-

	A	B
mean	3100 µg/g	
std. dev.	130 µg/g	
R.S.D.	4.2 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	0.14	10	5.7
mean	12	35	68

W 5 µg/g T 25 µg/g

CONTROL LIMITS: µg/g

Control		W.L. (x ±2σ)	R.L. (x±3σ)
QCRS85-1	L.L.	2800	2700
	U.L.	3400	3500

REMARKS:- Determination of boron MUST be on Atom Scan 2400 only.
- Extraction % (±) based on average recovery of 4 samples from Soil Research Institute - SO1, SO2, SO3 & SO4.
- Soils rich in organic matter should be ashed (like vegetation) prior to digestion.
- Samples requiring only Mg, Al and/or Ca are analyzed by Atom Scan.

SUMMARY REPORT OF QUALITY CONTROL DATA

ZINC IN SOIL

Operating Range =1.0000to 100.0 ug/g

IN-RUN DUPLICATES

Range	<1.0000	1.0000to20.00	20.00 to50.00	50.00 to100.0	>100.0
no.	7	1	24	19	17
s.w.		0.1414	10.5381	5.7089	
mean		12.5147	34.9634	67.6863	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
QCRS85-1	132	3084.898	129.4360	4.20

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
rb	110	0.177	0.6054

DATE 87/03/31

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: ZINC TEST CODE: ZNUT SAMPLE TYPE: SOIL
UNIT: Vegetation/Soil/Sediment SUPERVISOR: L. Pastorek

METHOD CODE:535BA0 -HMPNSOIL - AAS
REVISION NO: Original DATE: 1980
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- Approximately 20 g
Container- Glass jars (4 oz) with Bakelite screw cap.
Preservative- None
Other- Samples are air-dried and ground to less than 45 mesh.

SAMPLE PREPARATION:Partial Extn.-Yes Total Extn.- % Extracted-85±5
Procedure- Weigh 0.500g sample into a calibrated digestion tube and add 1.5 ml HNO_3 . Mix and stand at room temperature for 2 hrs. Add 4.5 ml HCl to each tube, mix and stand at room temperature for another hour. Digest in a hot block for 1 hour at 40°C, 1 hour at 60°C 1 hour at 80°C and 4 hrs at 100°C allowing 1/2 hr. ramping times
NOTE: The final volume should be less than 2 ml; if more, continue heating at 125°C to accomplish this.
Add 0.5 ml 5% KCl, dilute with DDW to 25 ml and mix thoroughly. Settle out suspended particles overnight and decant clear supernatant into disposable plastic tubes for analysis. Samples requiring the determination of < 5 elements and/or Na, K or Ag are analyzed by AAS.
INTERFERENCES: Several, which may be compensated for by background correction.
REPORTING RESULTS: Two significant figures. (µg/g Zn)
INSTRUMENTATION: PE 5000 atomic absorption spectrophotometer, PE Autosampler 50 and a PE Automatic Burner Controller,interfaced with Commodore Pet 4032, Tractor Printer 4022P & CBM 805C dual drive.
Calibration Range: 0 - 5 mg/l.
Resolution: 0.01 mg/l.
Sensitivity: conc. for absorbance of approx. 0.20 units = 1.0 mg/l
Instrument Detection Limit: 0.02 mg/l.
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 1.0 to 250 µg/g.
Accuracy- Not determined (ND).
Precision of Controls-

	A	B
mean	3400 µg/g	
std. dev.	150 µg/g	
R.S.D.	4.5 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.		7.0	7.1
mean		65	200

W 1 µg/g T 5 µg/g

CONTROL LIMITS: µg/g

	W.L. (x ±2σ)	R.L. (x ±3σ)
L.L.	3100	3000
U.L.	3700	3800

REMARKS: Control QCRS85-1
Extraction % (±) based on average recovery of 4 samples from Soil Research Institute - S01, S02, S03 & S04.

SUMMARY REPORT OF QUALITY CONTROL DATA

ZINC-AA IN SOIL

Operating Range =1.0000to 250.0 ug/g

IN-RUN DUPLICATES

Range	<1.0000	1.0000to50.00	50.00 to125.00	125.00to250.0	>250.0
no.	0	0	2	1	1
s.w.		0.0000	7.0180	7.0710	
mean		0.0000	64.7500	205.0000	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
S85-1	9	3366.670	150.0000	4.46

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.

DATE 87/04/23

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: CHLORIDE TEST CODE: CLEW SAMPLE TYPE:SOIL
UNIT: Vegetation/Soil/Sediment SUPERVISOR: L. Pastorek

METHOD CODE:305AI5 (CLSOILS)
REVISION NO: 1 DATE: Sept.,1987
NATURE OF LAST REVISION: From alkaline fusion to water extraction.

SAMPLE HANDLING:
Quantity Required- Approximately 20 g
Container-Glass jars (4 oz) with bakelite screw cap.
Preservative- None
Other-Samples are air dried and ground to less than 45 mesh

SAMPLE PREPARATION:Partial Extn.-Yes Total Extn.- % Extracted-
Procedure- Place 3.0g of air-dried sample in a 50ml centrifuge tube. Add 30 ml distilled water and shake for 1 hour on a reciprocating shaker. Centrifuge, filter through a membrane filter and analyze for water-extractable chloride by ion chromatography.

INTERFERENCES:

REPORTING RESULTS: Two significant figures (µg/g Cl)
INSTRUMENTATION: Dionex Ion Chromatograph 4000i, with 4270 Integrator, Autosampler, and Chart Recorder.

Calibration Range: 0 to 20 mg/l.
Resolution: 0.01 mg/l
Sensitivity:
Instrument Detection Limit: ND

PERFORMANCE CHARACTERISTICS:
Routine Operating Range-.01 to 200 µg/g. (based on 3 g sample).
Accuracy- Not determined (ND).
Precision of Controls-

	A	B
mean	15 µg/g	
std. dev.	3.2 µg/g	
R.S.D.	21 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	1.8	NA	NA
mean	9.7	NA	NA

W 1 µg/g T 5 µg/g

CONTROL LIMITS: µg/g

Control		W.L. (x ±2σ)	R.L. (x ±3σ)
	L.L.	8.6	5.4
	U.L.	21	25

REMARKS:- Three controls - A, B, & C.
- > ± 3 sd (standard deviations) on digested control samples before rejection of run.

SUMMARY REPORT OF QUALITY CONTROL DATA

EXCHLORIDE IN SOIL

Operating Range =0.0100to 100.0 ug/g

IN-RUN DUPLICATES

Range	<0.0100	0.0100to20.00	20.00 to50.00	50.00 to100.0	>100.0
no.	0	10	5	1	4
s.w.		1.7700	2.1500	0.0000	
mean		9.7100	33.2000	54.0000	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
clay	13	14.900	3.1780	21.33

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
blk	0	0.000	0.0000

DATE 87/04/03

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: FLUORIDE TEST CODE: FFUT SAMPLE TYPE:SOIL
UNIT: Vegetation/Soil/Sediment SUPERVISOR: L. Pastorek

METHOD CODE:301AIE - FLUORSED
REVISION NO: Original DATE: 1983, Sept.,1987
NATURE OF LAST REVISION: Update sheets.

SAMPLE HANDLING:
Quantity Required- Approximately 15 g
Container-SED-Glass jar with bakelite cap. SOIL-Wheaton Scint. vials
Preservative- None
Other-Samples are air dried and ground to less than 45 mesh

SAMPLE PREPARATION:Partial Extn.-Yes Total Extn.- % Extracted-
Procedure- Extract an air-dried sample overnight with 0.1N HClO4 in a water bath at 80°C. Add buffer TISAB III to reduce the formation of FH and FH2 and complex di- and trivalent cation interferences. Shake for one hour, cool and analyze by ion selective electrode.

INTERFERENCES: High concentrations of Al (> 500 µg/g).

REPORTING RESULTS: Two significant figures -(µg/g F)
INSTRUMENTATION: Orion Research Model 901; microprocessor Ionalyzer with Orion 9609 combination fluoride electrode.

Calibration Range: 0 - 5 µg/l.
Resolution: 0.01 µg/l).
Sensitivity:
Instrument Detection Limit: 0.01 µg/l.

PERFORMANCE CHARACTERISTICS:
Routine Operating Range-c.2 to 500 µg/g.
Accuracy- Not determined (ND).
Precision of Controls-

	A	B
mean	154 µg/g	20
std. dev.	7 µg/g	2.1
R.S.D.	5 %	11 %

Precision of Duplicates-

	low range	mid range	high range
s.d.	0.72	NA	NA
mean	27	NA	NA

W 0.5 µg/g T 2.5 µg/g

CONTROL LIMITS: µg/g

Control		W.L. (x ±2σ)	R.L. (x ±3σ)
	L.L.	16	14
	U.L.	24	26

REMARKS:- Three controls - A, B, & C.
- > ± 3 sd (standard deviations) on digested control samples before rejection of run.

SUMMARY REPORT OF QUALITY CONTROL DATA

FLUORIDE IN SOIL

Operating Range =0.2000to 500.0 ug/g

IN-RUN DUPLICATES

Range	<0.2000	0.2000to100.00	100.00to250.00	250.00to500.0	>500.0
no.	0	16	0	0	0
s.w.		0.7170	0.0000	0.0000	
mean		26.9300	0.0000	0.0000	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
veggc	6	154.500	7.2590	4.70
soilqc	6	19.550	2.0790	10.63
REF#3	4	41.500	2.9500	7.11

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
blk	0	0.000	0.0000

DATE 87/04/23

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME:NITROGEN-TOTAL TEST CODE:NNTKUR SAMPLE TYPE:SOIL
UNIT: Vegetation/Soil/Sediment SUPERVISOR: L. Pastorek

METHOD CODE:314CC2 TYPE:Acid digestion/automated colourimetry
REVISION NO: #1 DATE: Sept. 1987.
NATURE OF LAST REVISION: Update of QC data.

SAMPLE HANDLING:
Quantity Required- Approximately 20 g.
Container- Glass jars (4 oz.) with bakelite screw cap.
Preservative- None.
Other- Sample are air-dried and ground to less 45 mesh.

SAMPLE PREPARATION:Partial Extn.- Total Extn.-Yes % Extracted-
Procedure- Digest .2 to .4 sample with 7 ml of H_2SO_4 for about 2 hrs until intense fuming occurs. Add 2 g potassium persulphate and take to fuming until the digestate is clear. Cool and make up digestate to 100 ml. Titrate a 25 ml aliquot with 6.25N NaOH to pH 12 and filter out precipitated metals.
Add 8 ml of 4% EDTA solution, adjust the pH with 10% H_2SO_4 to the methyl red end point (pH 4) and adjust the volume to 100 ml. Analyze for nitrogen using the automated phenate method.

INTERFERENCES:

REPORTING RESULTS: Two signifcant figures (mg/g N)
INSTRUMENTATION: Automated colourimetric system (Technicon or equivalent) with 630 nm filters.

Calibration Range: 0 to 10 mg/L NH_3 as N.
Resolution: 0.01 mg/L.
Sensitivity:
Instrument Detection Limit: 0.1 mg/L.

PERFORMANCE CHARACTERISTICS:
Routine Operating Range-0.02 to 5 mg/g N dry wt. (for .2 g sample)
Accuracy- ND
Precision of Controls-

	A	B
mean	1.2 mg/g	
std. dev.	0.06 mg/g	
R.S.D.	5 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	0.17	0.30	0.82
mean	1.68	6.05	3.5

W .1 mg/g T .5 mg/g

CONTROL LIMITS: mg/g

Control		W.L. (x ±2σ)	R.L. (x±3σ)
	L.L.	1.1	1.0
	U.L.	1.3	1.4

REMARKS:- Control limits:

- Precision based on 0.2 g sample.
- Total phosphorus (PPUT) also on the same sample digest.
- Precision of duplicates - other values for sediments.

SUMMARY REPORT OF QUALITY CONTROL DATA

NITROGEN IN SOIL

Operating Range =0.0200to 5.0 mg/g

IN-RUN DUPLICATES

Range	<0.0200	0.0200to1.00	1.00 to2.50	2.50 to5.0	>5.0
no.	0	7	10	7	10
s.w.		0.0540	0.0820	0.2800	
mean		0.5500	1.8000	3.8400	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
refa	36	1.178	0.0582	4.94

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLK	0	0.000	0.0000

DATE 87/02/02

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME:PHOSPHORUS-TOT TEST CODE: PPUT SAMPLE TYPE:SOIL
UNIT: Vegetation/Soil/Sediment SUPERVISOR: L. Pastorek

METHOD CODE:314CC2 TYPE:Acid digestion/automated colourimetry
REVISION NO: Original #1 DATE: 1970 Sept. 1987.
NATURE OF LAST REVISION: Update of control limits

SAMPLE HANDLING:
Quantity Required- Approximately 20 g.
Container-Glass jars with bakelite screw cap.(4 oz.)
Preservative- None
Other- Samples are air-dried and ground to less than 45 mesh.

SAMPLE PREPARATION:Partial Extn.- Total Extn.-Yes % Extracted-
Procedure-Digest .2 to .4g sample with 7 ml of H_2SO_4 for about 2 hrs until intense fuming occurs. Add 2g potassium persulphate and take to fuming until the digestate is clear. Cool and make up digestate to 100 ml. Titrate a 25 ml aliquot with 6.25N NaOH to pH 12 and filter out precipitated metals.
Add 8 ml of 4% EDTA solution, adjust the pH with 10% H_2SO_4 to the methyl red end point (pH 4) and adjust the volume to 100 ml. Analyze phosphorus using the automated stannous chloride reduced phosphomolybdate procedure.

INTERFERENCES: Heavy metal interferences removed by precipitated filtration.
REPORTING RESULTS: Two significant figures (mg/g P)
INSTRUMENTATION: Automated colourimetric system (Technicon or equivalent) with 660 nm filters.

Calibration Range: 0 to 2 mg/l PO_4 as P.
Resolution: 0.002 mg/l.
Sensitivity:
Instrument Detection Limit: 0.02 mg/l.

PERFORMANCE CHARACTERISTICS:
Routine Operating Range-.005 to 1 mg/g PO_4 as P dry wt.(for 0.2 g)
Accuracy-
Precision of Controls-

	A	B
mean	0.891mg/g	
std. dev.	0.031mg/g	
R.S.D.	3 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	0.04	0.05	0.04
mean	0.53	1.18	2.42

W .02 mg/g T .10 mg/g

CONTROL LIMITS: mg/g

Control		W.L. (x ±2σ)	R.L. (x ±3σ)
	L.L.	0.83	0.80
	U.L.	0.95	0.98

REMARKS:- Control limits:

- Precision based on 0.2 g sample.

SUMMARY REPORT OF QUALITY CONTROL DATA

PHOSPHORUS IN SOIL

Operating Range =0.0050to 1.0 mg/g

IN-RUN DUPLICATES

Range	<0.0050	0.0050to0.20	0.20 to0.50	0.50 to1.0	>1.0
no.	1	0	5	12	15
s.w.		0.0000	0.0170	0.0450	
mean		0.0000	0.3700	0.7100	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
refa	36	0.887	0.0306	3.45

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLK	0	0.000	0.0000

DATE 87/02/02

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME:SULPHATE TEST CODE:SSO4UR SAMPLE TYPE:SOIL
UNIT:Veg./Soil/Sediment SUPERVISOR:L. Pastorek

METHOD CODE:300BI5
REVISION NO: DATE: June 1885
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- Approximately 20 g
Container- Glass jar (4 oz.) with bakelite screw cap
Preservative- None
Other- Samples are air-dried and ground to less than 45 mesh.

SAMPLE PREPARATION:Partial Extn.-Yes Total Extn.- % Extracted-
Procedure- Sample (3 g) is shaken with 30 ml deionized distilled water for 1 hour in a capped centrifuge tube. Sample is centrifuged for 10 minutes at 5000 rpm. Sample is filtered through a membrane filter and analyzed for water-extractable sulphate by ion chromatograph.

INTERFERENCES:

REPORTING RESULTS:ug/g SO4 to two significant figures.
INSTRUMENTATION:Dionex Ion Chromatograph 4000i, with 4270 Integrator, Autosampler, and Chart Recorder.

Calibration Range: 0 to 20 mg/L
Resolution:.0.01 mg/L
Sensitivity:
Instrument Detection Limit:
PERFORMANCE CHARACTERISTICS:
Routine Operating Range-.01 to 200 ug/g
Accuracy-
Precision of Controls-

		A	B
	mean	72.1 ug/g	43.8 ug/g
	std. dev.	21.9 ug/g	11.1 ug/g
	R.S.D.	30 %	25 %

Precision of Duplicates-

	low range	mid range	high range
s.d.	ND	ND	ND
mean	ND	ND	ND

W 10 µg/g T 50 µg/g

CONTROL LIMITS:

Control	µg/g	W.L. (x ±2σ)	R.L. (x ±3σ)
	L.L.	96	74
	U.L.	180	210

REMARKS: Changes in sediment sol'n ratio used may cause differences in amount of sulphate recovered. A standard ratio should be used if data is being compared.

SUMMARY REPORT OF QUALITY CONTROL DATA

SULPHATE IN SOIL

Operating Range =0.0100to 100.0 ug/g

IN-RUN DUPLICATES

Range	<0.0100	0.0100to20.00	20.00 to50.00	50.00 to100.0	>100.0
no.	0	0	0	2	4
s.w.		0.0000	0.0000	0.0000	
mean		0.0000	0.0000	63.0000	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
soil	4	140.600	31.5330	22.43
clay	4	72.110	21.8650	30.32
sedqc	4	43.830	11.0580	25.23

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
blk	0	0.000	0.0000

DATE 87/04/22

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: SULPHUR TEST CODE: SSUT SAMPLE TYPE: SOIL
UNIT: Vegetation/Soil/Sediment SUPERVISOR: L. Pastorek

METHOD CODE:003AF7 (LECOS)
REVISION NO: Original DATE: 1980
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- Approximately 20 g
Container- Glass jars (4 oz) with bakelite screw cap.
Preservative- None
Other- Samples are air-dried and ground to less than 45 mesh.

SAMPLE PREPARATION:Partial Extn.- Total Extn.-yes % Extracted-84±4%
Procedure- Weigh 0.250 g sample into a ceramic crucible. Spread evenly over sample one scoop of copper accelerator and two scoops of iron chip accelerator. Cover crucible and ignite in induction furnace at 1600°C for 7 minutes in a continuous flow of oxygen. S is oxidized to SO2 (and some SO3). Titrate with KIO2 in a dilute HCl solution using starch indicator. The end point detection is electronically set and controlled. Readings are directly taken from the burette and blk subtracted. A factor is used if necessary.

INTERFERENCES:High concs. fluorides, chlorides and organics (carbon).

REPORTING RESULTS: Two significant figures. (% S)
INSTRUMENTATION: Leco induction furnace (HF 10) and a Leco automatic titrator.

Calibration Range: 0 - 0.035 %.
Resolution: 0.001 %.
Sensitivity: ND
Instrument Detection Limit: 0.001 %.

PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.001 % to 0.100 %.
Accuracy- 95.7 % (based on calibration standards).
Precision of Controls-

	A	B
mean	0.040 %	
std. dev.	0.0043 %	
R.S.D.	10.8 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	0.0011	0.0023	0.0045
mean	0.0114	0.031	0.067

W 0.001 % T 0.005 %

CONTROL LIMITS:%

	W.L. (x ±2σ)	R.L. (x±3σ)
L.L.	0.031	0.027
U.L.	0.049	0.053

REMARKS: Control S-64
Extraction % (±) based on average of 2 standard reference materials.

SUMMARY REPORT OF QUALITY CONTROL DATA

SULPHUR IN SOIL

Operating Range =0.0010to 0.1 %

IN-RUN DUPLICATES

Range	<0.0010	0.0010to0.02	0.02 to0.05	0.05 to0.1	>0.1
no.	0	30	44	14	9
s.w.		0.0011	0.0023	0.0045	
mean		0.0114	0.0313	0.0674	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
qcrs85-1	139	0.043	0.0036	8.39

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.

DATE 87/04/22